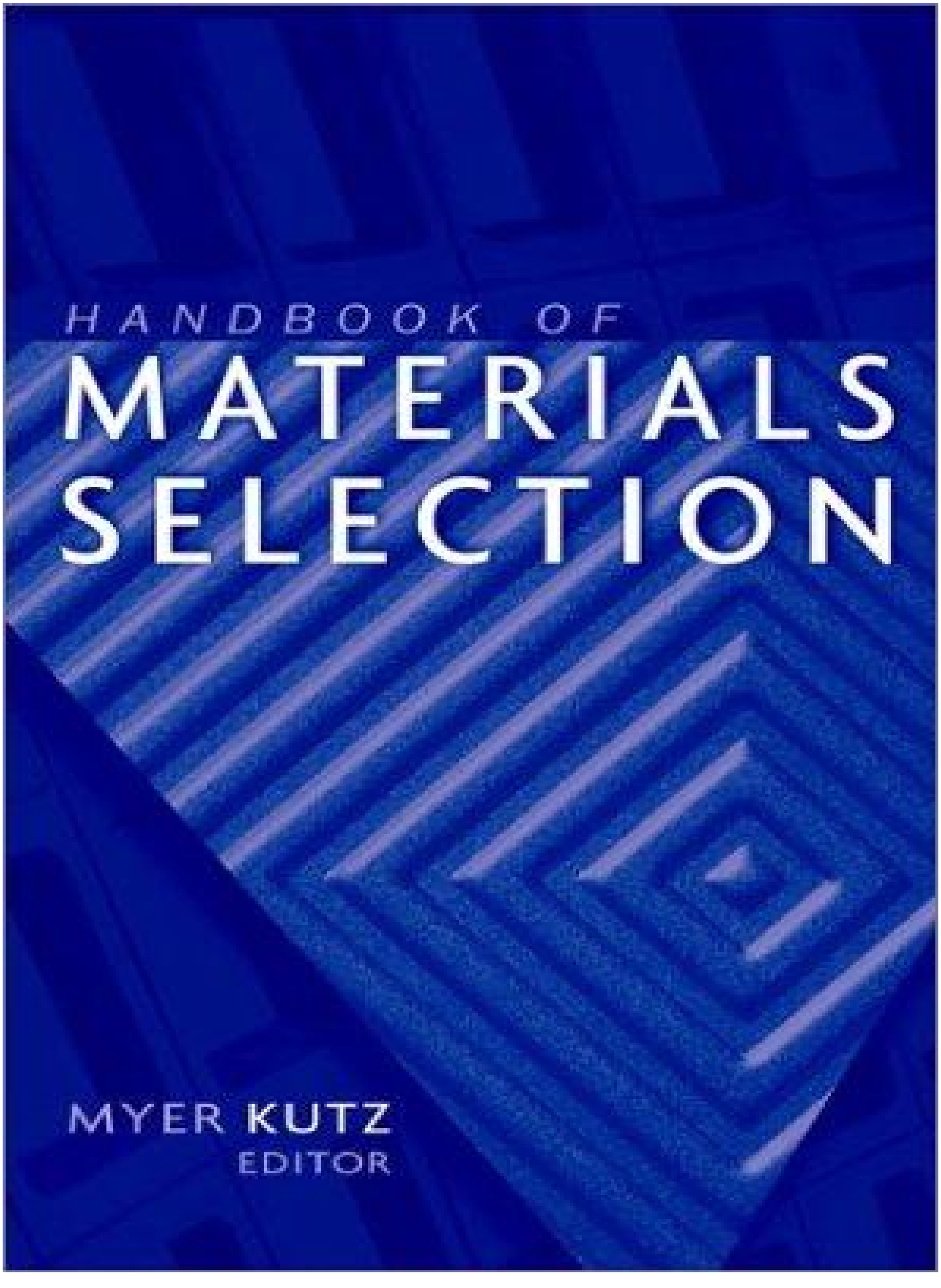

HANDBOOK OF
MATERIALS SELECTION

MYER KUTZ
EDITOR

Handbook of Materials Selection

Handbook of Materials Selection. Edited by Myer Kutz

Handbook of Materials Selection

Edited by

MYER KUTZ
Myer Kutz Associates, Inc.

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To Merrilyn, Bill, and David
The Future Is Yours

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PREFACE

Invention is often born of the need, or just the desire, to improve something. This simple statement (a restatement, in a way, of the old saw ‘‘necessity is the mother of invention’’) is the driving force behind the development of the *Handbook of Materials Selection*. The audience for this handbook consists of practicing engineers and the people who work with them, all of whom need to determine what materials they might specify, order, and use to make something better, whether it’s a dental implant, an electronic package, an airplane, or a highway overpass. The choices are not always as clear cut, nor are they as straightforward, as they once were. In the past, one material (e.g., steel) or a class of materials (e.g., metals) might have been all an engineer would have needed to consider for a particular application. But now different classes of materials compete for consideration, in order that a manufactured part or assembly be as inexpensive, or as light, or as long-lasting as possible, to name just a few factors that might have to be taken into account. So whereas an engineer might have turned in the past to a single supplier’s materials properties tables to make a selection, now he or she might first turn to the engineer’s most trusted information source—colleagues, whose collective expertise can be brought to bear on an improved materials selection procedure.

So an important purpose of a publication such as this handbook is to assemble a collection of experts to provide advice to an engineer. If a handbook is to do this job effectively, its assigned experts should have a wide range of professional experience. They should have worked in a variety of settings. In keeping with this concept, the *Handbook of Materials Selection* is the product of the efforts of over 50 contributors who have experience in five different environments: a little over 40 percent are from mainly industrial backgrounds; a little under 30 percent are U.S. university faculty members, many with some experience in industry, while another 10 percent are, or have been until recently, on the faculty of academic institutions in Egypt, Israel, Germany, and England; the rest work at U.S. government installations or at research institutes, both private and university affiliated.

Whatever their background and experience may be, the contributors to this handbook have written in a style that reflects practical discussion informed by real-world experience. The intent is for readers to feel that they are in the presence of experienced engineers, materials specialists, teachers, researchers, and consultants who are well acquainted with the multiplicity of issues that govern the selection of materials for industrial applications. At the same time, the level is such that students and recent graduates can find the handbook as accessible as experienced engineers.

As much as practicable, contributors have employed visual displays, such as tables, charts, and photographs, to illustrate the points they make and the examples they draw on. They have discussed current trends in the specification, availability, and use of materials. Also, wherever appropriate, discussion enables readers to look into the near-term future.

Nevertheless, no information resource, I mean no handbook, no shelf of books, not even a web site or a portal on the Internet (not yet, at least), can hope to inspire every new product whose successful introduction and long service life are predicated on innovative and adroit materials selection, much less inspire every new version of existing products that is cheaper, lighter, or flashier than its predecessors because of a clever material substitution. Why, then, develop a one-volume *Handbook of Materials Selection*? The powerful premise driving this 1,500-page handbook is that, in terms of materials selection, what works now, as well as what has failed in the past, can serve as an experiential platform on which practicing engineers can employ the modern multidisciplinary training they now receive.

My intention has been to create a practical reference for engineers wanting to explore questions about selecting materials for specific industrial applications. In my view, there are two sets of useful questions worth exploring. One set covers practical examples of the what, why, and how of materials selection:

What materials have been used in particular industrial applications?
Why were these materials selected?
Were the materials processed in special ways?
How did material properties relate to performance in service?
Were there any problems initially, and did any develop later?
What precautions are recommended?
What were the key tradeoffs between properties and performance?
What were the limitations imposed by the selected materials?

A second set of questions relates to a practicing engineer's particular design situation:

What materials might have the characteristics that meet the needs of the application I'm working on?
Where would I find information about such materials?
What processing techniques might I use to create parts or components from these materials?
How do I take into account properties and manufacturing processes in the design process?
How would I confirm that the materials I specify and purchase have the properties I'm looking for?
How does the organization I'm working for go about supplying the materials required by the design I'm proposing, and what limitations may be imposed on my selection by such factors as cost, environmental degradation, etc.?

The emphasis in the handbook is on practical issues rather than on basic science, on design and manufacturability issues, on where to find properties information, much of which is now electronic, and on instructive applications and case studies where engineers have taken advantage of distinctive properties offered by different classes of materials. Metals, nonmetallic materials, including plastics and ceramics, and composites get equal coverage, as appropriate.

In order to answer such questions as the ones I have posed above, I arranged the contents of the handbook in seven parts. The first part, just one chapter long, but important nonetheless and a good introduction to the field of materials selection, is on quantitative methods that a practitioner can apply to materials selection problems. The second part covers the range of major materials—metallic, nonmetallic, and composite, from the tried and true to the new and novel—that engineers use nowadays to make things. A couple of these chapters deal specifically with the potential problems that practitioners should be aware of when selecting particular materials.

The third part of the handbook covers sources of materials data, including a librarian's advice on finding, as well as evaluating the reliability of, such data, methods for managing the data that an organization has acquired, and how the data are used for procuring materials. Once you've obtained a material, what exactly do you have? The fourth part of the handbook deals with the issue of testing—what equipment is used to determine the properties of the different classes of materials, what standards govern test procedures, and what organizations are in the business of providing testing services.

What about the life expectancy of the thing you've designed and made from the material you selected? Another important factor in materials selection is knowing how different classes of materials fail, which is the subject of the chapters that comprise the fifth part of the handbook. The final aspect of materials selection involves knowing about the manufacturing processes used to make things from available classes of materials, which is the subject of the chapters that make up the sixth part of the handbook.

The handbook's last, and largest, section, which sets it apart from other handbooks in the materials field, includes 11 chapters that deal not only with a broad range of industrial applications, but also with design and assembly issues involved in using composites and plastics, as well as chapters on materials that provide improved wear resistance. The applications chapters cover aerospace, medical, electronic, telecommunications, sports, and construction industries.

A few chapters in this handbook, which are not more than a few years old, have been repurposed from the second edition of another Wiley publication that I have developed, the *Mechanical Engineers' Handbook*. For the most part, however, the contributions in the *Handbook of Materials Selection* were cooked to order, so to speak. All of them are miracles, and I am eternally grateful to the busy men and women who took the time and trouble to write them.

My thanks to Wiley's internal and external production personnel for their speed and diligence. They, too, are in the business of making something better. Special thanks to my acquiring editor, Bob Argentieri, who shepherded the project through the corporate labyrinth. Not long after I drove down to Manhattan with the handbook manuscript in file folders in two cartons on the back seat of my car, Bob and his wife, Anne, had their third child. She will grow up in a world changed and improved by the materials-selection decisions that engineers make every day. I hope this handbook helps to make some of those decisions the best that they can be.

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PART 1

QUANTITATIVE METHODS OF MATERIALS SELECTION

Handbook of Materials Selection. Edited by Myer Kutz

CHAPTER 1

QUANTITATIVE METHODS OF MATERIALS SELECTION

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1 INTRODUCTION

It is estimated that there are more than 40,000 currently useful metallic alloys and probably close to that number of nonmetallic engineering materials such as plastics, ceramics and glasses, composite materials, and semiconductors. This large number of materials and the many manufacturing processes available to the engineer, coupled with the complex relationships between the different selection parameters, often make the selection of a materials for a given component a difficult task. If the selection process is carried out haphazardly, there will be the risk of overlooking a possible attractive alternative material. This risk can be reduced by adopting a systematic material selection procedure. A variety of quantitative selection procedures have been developed to analyze the large amount of data involved in the selection process so that a systematic evaluation

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Handbook of Materials Selection. Edited by Myer Kutz

can be made.[1–11] Several of the quantitative procedures can be adapted to use computers in selection from a data bank of materials.[12–15]

Experience has shown that it is desirable to adopt the holistic decision-making approach of concurrent engineering in product development in most industries. With concurrent engineering, materials and manufacturing processes are considered in the early stages of design and are more precisely defined as the design progresses from the concept to the embodiment and finally the detail stages. Figure 1 defines the different stages of design and shows the related activities of the material and manufacturing process selection. The figure illustrates the progressive nature of materials and process selection and defines three stages of selection—namely initial screening, developing and comparing alternatives, and selecting the optimum solution. Sections 2, 3, and 4 of this chapter discuss these three stages of material and process selection in more detail, and Section 5 gives a case study to illustrate the procedure.

Although the materials and process selection is often thought of in terms of new product development, there are many other incidents where materials substitution is considered for an existing product. Issues related to material substitution are discussed in Section 6 of this chapter.

Unlike the exact sciences, where there is normally only one single correct solution to a problem, materials selection and substitution decisions require the consideration of conflicting advantages and limitations, necessitating compromises and trade-offs; as a consequence, different satisfactory solutions are possible. This is illustrated by the fact that similar components performing similar functions, but produced by different manufacturers, are often made from different materials and even by different manufacturing processes.

2 INITIAL SCREENING OF MATERIALS

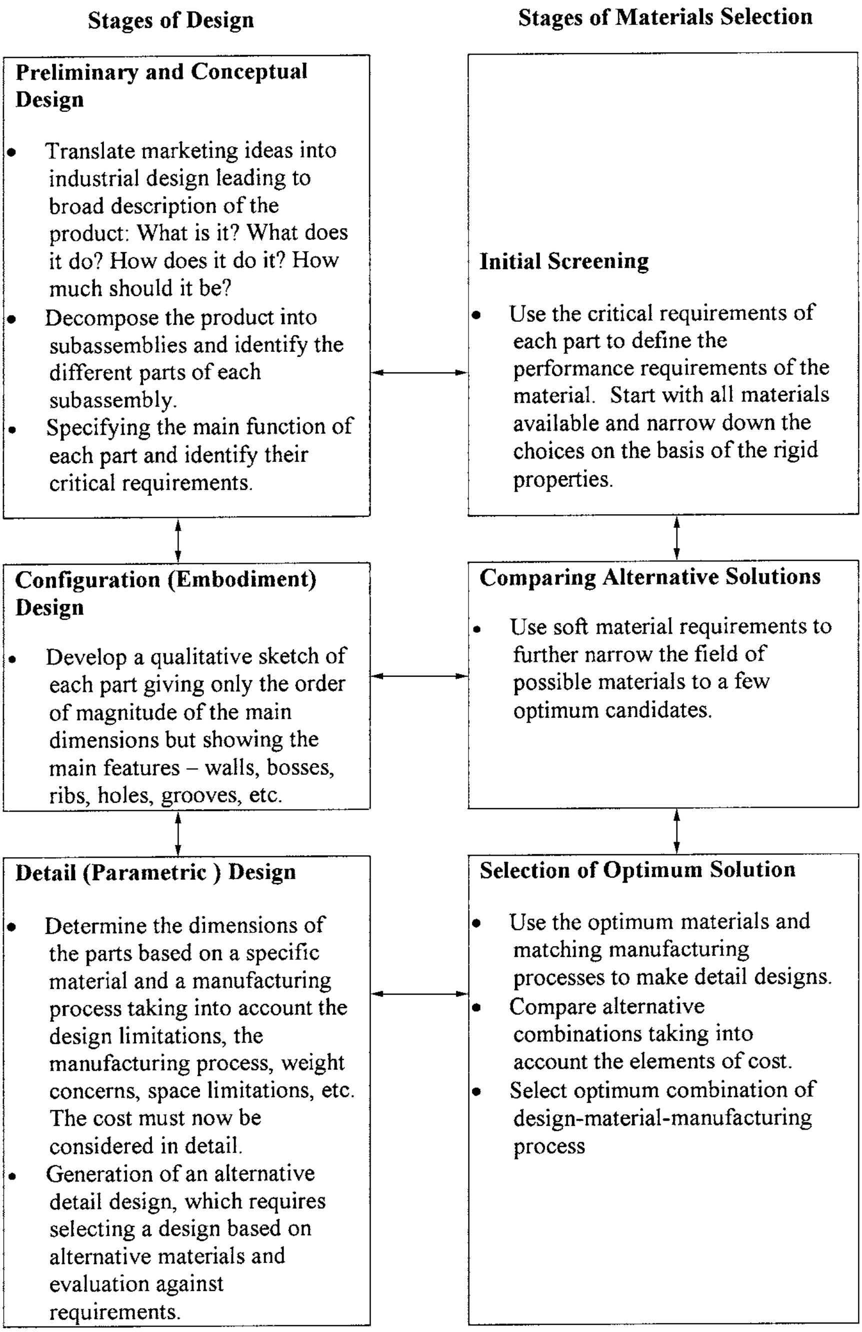
In the first stages of development of a new product, the following questions may be posed: What is it? What does it do? How does it do it? To answer these questions it is necessary to specify the performance requirements of the different parts involved in the design and to broadly outline the main materials performance and processing requirements. This allows the initial screening of materials whereby certain classes of materials and manufacturing processes may be eliminated and others chosen as likely candidates.

2.1 Analysis of Material Performance Requirements

The material performance requirements can be divided into five broad categories, namely functional requirements, processability requirements, cost, reliability, and resistance to service conditions.[1]

Functional Requirements

Functional requirements are directly related to the required characteristics of the part or the product. For example, if the part carries a uniaxial tensile load, the yield strength of a candidate material can be directly related to the load-carrying capacity of the product. However, some characteristics of the part or product may not have simple correspondence with measurable material properties, as in the case of thermal shock resistance, wear resistance, reliability, etc. Under these conditions, the evaluation process can be quite complex and may depend upon



Stages of Design

Stages of Materials Selection

Preliminary and Conceptual Design

- Translate marketing ideas into industrial design leading to broad description of the product: What is it? What does it do? How does it do it? How much should it be?
- Decompose the product into subassemblies and identify the different parts of each subassembly.
- Specifying the main function of each part and identify their critical requirements.

Initial Screening

- Use the critical requirements of each part to define the performance requirements of the material. Start with all materials available and narrow down the choices on the basis of the rigid properties.

Configuration (Embodiment) Design

- Develop a qualitative sketch of each part giving only the order of magnitude of the main dimensions but showing the main features – walls, bosses, ribs, holes, grooves, etc.

Comparing Alternative Solutions

- Use soft material requirements to further narrow the field of possible materials to a few optimum candidates.

Detail (Parametric) Design

- Determine the dimensions of the parts based on a specific material and a manufacturing process taking into account the design limitations, the manufacturing process, weight concerns, space limitations, etc. The cost must now be considered in detail.
- Generation of an alternative detail design, which requires selecting a design based on alternative materials and evaluation against requirements.

Selection of Optimum Solution

- Use the optimum materials and matching manufacturing processes to make detail designs.
- Compare alternative combinations taking into account the elements of cost.
- Select optimum combination of design-material-manufacturing process

Fig. 1 Stages of design and the related stages of materials selection.

predictions based on simulated service tests or upon the most closely related mechanical, physical, or chemical properties. For example, thermal shock resistance can be related to thermal expansion coefficient, thermal conductivity, modulus of elasticity, ductility, and tensile strength. On the other hand, resistance to stress corrosion cracking can be related to tensile strength, K_{ISCC}, and electrochemical potential.

Processability Requirements

The processability of a material is a measure of its ability to be worked and shaped into a finished part. With reference to a specific manufacturing method, processability can be defined as castability, weldability, machinability, etc. Ductility and hardenability can be relevant to processability if the material is to be deformed or hardened by heat treatment, respectively. The closeness of the stock form to the required product form can be taken as a measure of processability in some cases.

It is important to remember that processing operations will almost always affect the material properties so that processability considerations are closely related to functional requirements.

Cost

Cost is usually an important factor in evaluating materials because in many applications there is a cost limit for a material intended to meet the application requirements. When the cost limit is exceeded, the design may have to be changed to allow for the use of a less expensive material. The cost of processing often exceeds the cost of the stock material. In some cases, a relatively more expensive material may eventually yield a less expensive product than a low-priced material that is more expensive to process.

Reliability Requirements

Reliability of a material can be defined as the probability that it will perform the intended function for the expected life without failure. Material reliability is difficult to measure because it is not only dependent upon the material's inherent properties, but it is also greatly affected by its production and processing history. Generally, new and nonstandard materials will tend to have lower reliability than established, standard materials.

Despite difficulties of evaluating reliability, it is often an important selection factor that must be taken into account. Failure analysis techniques are usually used to predict the different ways in which a product can fail and can be considered as a systematic approach to reliability evaluation. The causes of failure of a part in service can usually be traced back to defects in materials and processing, to faulty design, unexpected service conditions, or misuse of the product.

Resistance to Service Conditions

The environment in which the product or part will operate plays an important role in determining the material performance requirements. Corrosive environments, as well as high or low temperatures, can adversely affect the performance of most materials in service. Whenever more than one material is involved in an application, compatibility becomes a selection consideration. In a thermal environment, for example, the coefficients of thermal expansion of all the materials involved may have to be similar in order to avoid thermal stresses. In wet environments, materials that will be in electrical contact should be chosen carefully to avoid galvanic corrosion. In applications where relative movement exists between different parts, wear resistance of the materials involved should be considered. The design should provide access for lubrication, otherwise self-lubricating materials have to be used.

2.2 Quantitative Methods for Initial Screening

Having specified the performance requirements of the different parts, the required material properties can be established for each of them. These properties may be quantitative or qualitative, essential or desirable. For example, the function of a connecting rod in an internal combustion engine is to connect the piston to the crank shaft. The performance requirements are that it should transmit the power efficiently without failing during the expected life of the engine. The essential material properties are tensile and fatigue strengths, while the desirable properties that should be maximized are processability, weight, reliability, and resistance to service conditions. All these properties should be achieved at a reasonable cost. The selection process involves the search for the material or materials that would best meet those requirements. The starting point for materials selection is the entire range of engineering materials. At this stage, creativity is essential in order to open up channels in different directions and not to let traditional thinking interfere with the exploration of ideas. A steel may be the best material for one design concept while a plastic is best for a different concept, even though the two designs provide the same function.

After all the alternatives have been suggested, the ideas that are obviously unsuitable are eliminated and attention is concentrated on those that look practical. At the end of this phase, quantitative methods can be used for initial screening in order to narrow down the choices to a manageable number for subsequent detailed evaluation. Following are some of the quantitative methods for initial screening of materials.

Limits on Material Properties

Initial screening of materials can be achieved by first classifying their performance requirements into two main categories[1]:

- Rigid, or go–no-go, requirements
- Soft, or relative, requirements

Rigid requirements must be met by the material if it is to be considered at all. Such requirements can be used for the initial screening of materials to eliminate the unsuitable groups. For example, metallic materials are eliminated when selecting materials for an electrical insulator. If the insulator is to be flexible, the field is narrowed further as all ceramic materials are eliminated. Other examples of the material rigid requirements include behavior under operating temperature, resistance to corrosive environment, ductility, electrical and thermal conductivity or insulation, and transparency to light or other waves. Examples of process rigid requirements include batch size, production rate, product size and shape, tolerances, and surface finish. Whether or not the equipment or experience for a given manufacturing process exist in a plant can also be considered as a hard requirement in many cases. Compatibility between the manufacturing process and the material is also an important screening parameter. For example, cast irons are not compatible with sheet metal forming processes and steels are not easy to process by die casting. In some cases, eliminating a group of materials results in automatic elimination of some manufacturing processes. For example, if plastics are eliminated because service temperature is too high, injection and transfer molding should be eliminated as they are unsuitable for other materials.

Soft, or relative, requirements are subject to compromise and trade-offs. Examples of soft requirements include mechanical properties, specific gravity, and cost. Soft requirements can be compared in terms of their relative importance, which depends on the application under study.

Cost per Unit Property Method

The cost per unit property method is suitable for initial screening in applications where one property stands out as the most critical service requirement.[1] As an example, consider the case of a bar of a given length (L) to support a tensile force (F). The cross-sectional area (A) of the bar is given by

$$A = F/S \tag{1}$$

where S = working stress of the material, which is related to its yield strength by an appropriate factor of safety.

The cost of the bar (C') is given by

$$C' = C\rho AL = (C\rho FL)/S \tag{2}$$

where C = cost of the material per unit mass
ρ = density of the material

Since F and L are constant for all materials, comparison can be based on the cost of unit strength, which is the quantity:

$$[(C\rho)/S] \tag{3}$$

Materials with lower cost per unit strength are preferable. If an upper limit is set for the quantity $[(C\rho)/S]$, then materials satisfying this condition can be identified and used as possible candidates for more detailed analysis in the next stage of selection.

The working stress of the material in Eqs. 1, 2, and 3 is related to the static yield strength of the material since the applied load is static. If the applied load is alternating, it is more appropriate to use the fatigue strength of the material. Similarly, the creep strength should be used under loading conditions that cause creep.

Equations similar to 2 and 3 can be used to compare materials on the basis of cost per unit stiffness when the important design criterion is deflection in the bar. In such cases, S is replaced by the elastic modulus of the material. The

Table 1 Formulas for Estimating Cost per Unit Property[1]

Cross Section and Loading Condition	Cost per Unit Strength	Cost per Unit Stiffness
Solid cylinder in tension or compression	$C\rho/S$	$C\rho/E$
Solid cylinder in bending	$C\rho/S^{2/3}$	$C\rho/E^{1/2}$
Solid cylinder in torsion	$C\rho/S^{2/3}$	$C\rho/G^{1/2}$
Solid cylindrical bar as slender column	—	$C\rho/E^{1/2}$
Solid rectangle in bending	$C\rho/S^{1/2}$	$C\rho/E^{1/3}$
Thin-walled cylindrical pressure vessel	$C\rho/S$	—

above equations can also be modified to allow comparison of different materials under loading systems other than uniaxial tension. Table 1 gives some formulas for the cost per unit property under different loading conditions based on either yield strength or stiffness.

Ashby's Method

Ashby's material selection charts[4,5,9,10] are also useful for initial screening of materials. Figure 2 plots the strength against density for a variety of materials. Depending upon the geometry and type of loading, different S–ρ relationships apply as shown in Table 1. For simple axial loading, the relationship is S/ρ. For solid rectangle under bending, $S^{1/2}/\rho$ applies, and for solid cylinder under bending or torsion the relationship $S^{2/3}/\rho$ applies. Lines with these slopes are shown in Fig. 2. Thus if a line is drawn parallel to the line $S/\rho = C$, all the materials that lie on the line will perform equally well under simple axial loading conditions. Materials above the line are better and those below it are worse. A similar diagram can be drawn for elastic modulus against density and formulas similar to those in Table 1 can be used to screen materials under conditions where stiffness is a major requirement

Dargie's Method

The initial screening of materials and processes can be a tedious task if performed manually from handbooks and supplier catalogs. This difficulty has prompted the introduction of several computer-based systems for materials and/or process selection.[12–15] As an illustrative example, the system (MAPS 1) proposed by Dargie et al.[15] will be briefly described here. For this system, Dargie et al. proposed a part classification code similar to that used in group technology.

The first five digits of the MAPS 1 code are related to the elimination of unsuitable manufacturing processes. The first digit is related to the batch size. The second digit characterizes the bulk and depends on the major dimension and whether the part is long, flat, or compact. The third digit characterizes the shape, which is classified on the basis of being prismatic, axisymmetric, cup shaped, nonaxisymmetric, and nonprismatic. The fourth digit is related to tolerance and the fifth digit is related to surface roughness

The next three digits of the MAPS 1 code are related to the elimination of unsuitable materials. The sixth digit is related to service temperature. The seventh digit is related to the acceptable corrosion rate. The eighth digit characterizes the type of environment to which the part is exposed.

The system uses two types of databases for preliminary selection:

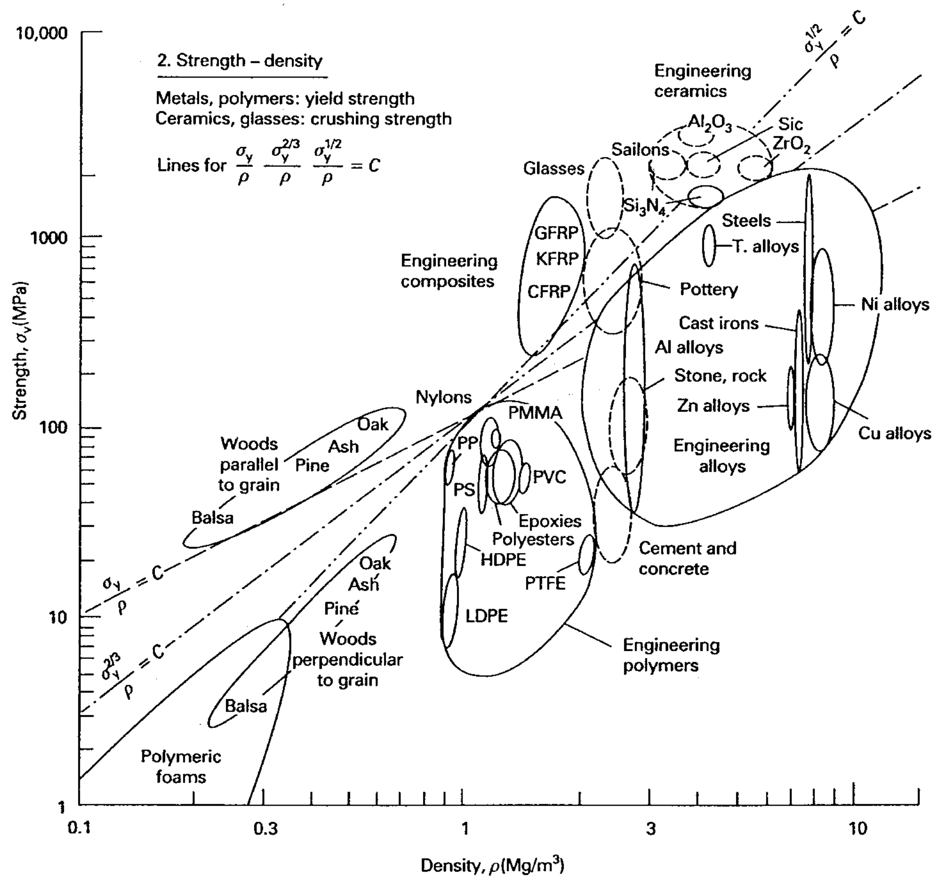


Fig. 2 Example of Ashby's materials selection charts (from Ref. 10, with permission from The Institute of Materials).

- Suitability matrices
- Compatibility matrix

The suitability matrices deal with the suitability of processes and materials for the part under consideration. Each of the code digits has a matrix. The columns of the matrix correspond to the value of the digit and the rows correspond to the processes and materials in the database. The elements of the matrix are either 0, indicating unsuitability, or 2 indicating suitability.

The compatibility matrix expresses the compatibility of the different combinations of processes and materials. The columns of the matrix correspond to the materials while the rows correspond to the processes. The elements of the matrix are either 0 for incompatible combinations, 1 for difficult or unusual combinations, or 2 for combinations used in usual practice.

Based on the part code, the program generates a list of candidate combinations of materials and processes to produce it. This list helps the designer to identify possible alternatives early in the design process and to design for ease of manufacture.

3 COMPARING ALTERNATIVE SOLUTIONS

After narrowing down the field of possible materials using one or more of the quantitative initial screening methods described in Section 2, quantitative methods can be used to further narrow the field of possible materials and matching manufacturing processes to a few optimum candidates that have good combinations of soft requirements. Several such methods are described in Refs. 1 and 2 and following is a description of one of the methods.

3.1 Weighted-Properties Method

In the weighted-properties method each material requirement, or property, is assigned a certain weight, depending on its importance to the performance of the part in service.[1] A weighted-property value is obtained by multiplying the numerical value of the property by the weighting factor (α). The individual weighted-property values of each material are then summed to give a comparative materials performance index (γ). Materials with the higher performance index (γ) are considered more suitable for the application.

Digital Logic Method

In the cases where numerous material properties are specified and the relative importance of each property is not clear, determinations of the weighting factors, α, can be largely intuitive, which reduces the reliability of selection. The digital logic approach can be used as a systematic tool to determine α.[1] In this procedure evaluations are arranged such that only two properties are considered at a time. Every possible combination of properties or goals is compared and no shades of choice are required, only a yes or no decision for each evaluation. To determine the relative importance of each property or goal a table is constructed, the properties or goals are listed in the left-hand column, and comparisons are made in the columns to the right, as shown in Table 2.

In comparing two properties or goals, the more important goal is given numerical one (1) and the less important is given zero (0). The total number of possible decisions $N = n\ (n - 1)/2$, where n is the number of properties or goals under consideration. A relative emphasis coefficient or weighting factor,

Table 2 Determination of Relative Importance of Goals Using Digital Logic Method[1]

	Number of Positive Decisions $N = n(n - 1)/2$											Relative Emphasis Coefficient
Goals	1	2	3	4	5	6	7	8	9	10	Positive Decisions	α
1	1	1	0	1							3	0.3
2	0				1	0	1				2	0.2
3		0			0			1	0		1	0.1
4			1			1		0		0	2	0.2
5				0			0		1	1	2	0.2
	Total number of positive decisions										10	$\Sigma\alpha = 1.0$

α, for each goal is obtained by dividing the number of positive decisions for each goal (m) into the total number of possible decisions (N). In this case $\Sigma\alpha = 1$.

To increase the accuracy of decisions based on the digital logic approach, the yes–no evaluations can be modified by allocating gradation marks ranging from 0 (no difference in importance) to 3 (large difference in importance). In this case, the total gradation marks for each selection criterion are reached by adding up the individual gradation marks. The weighting factors are then found by dividing these total gradation marks by their grand total.

Performance Index

In its simple form, the weighted-properties method has the drawback of having to combine unlike units, which could yield irrational results. This is particularly true when different mechanical, physical, and chemical properties with widely different numerical values are combined. The property with higher numerical value will have more influence than is warranted by its weighting factor. This drawback is overcome by introducing scaling factors. Each property is so scaled that its highest numerical value does not exceed 100. When evaluating a list of candidate materials, one property is considered at a time. The best value in the list is rated as 100 and the others are scaled proportionally. Introducing a scaling factor facilitates the conversion of normal material property values to scaled dimensionless values. For a given property, the scaled value, B, for a given candidate material is equal to:

$$B = \text{Scaled property} = \frac{\text{Numerical value of prperty} \times 100}{\text{Maximum value in the list}} \tag{4}$$

For properties such as cost, corrosion or wear loss, weight gain in oxidation, etc., a lower value is more desirable. In such cases, the lowest value is rated as 100 and B is calculated as:

$$B = \text{Scaled property} = \frac{\text{Minimum value in the list} \times 100}{\text{Numerical value of property}} \tag{5}$$

For material properties that can be represented by numerical values, application of the above procedure is simple. However, with properties such as corrosion and wear resistance, machinability and weldability, etc., numerical values are rarely given and materials are usually rated as very good, good, fair, poor, etc. In such cases, the rating can be converted to numerical values using an arbitrary scale. For example, corrosion resistance rating—excellent, very good, good, fair, and poor—can be given numerical values of 5, 4, 3, 2, and 1, respectively. After scaling the different properties, the material performance index (γ) can be calculated as:

$$\text{Material performance index} = \gamma = \sum_{i=1}^{n} B_i\alpha_i \tag{6}$$

where i is summed over all the n relevant properties.

Cost (stock material, processing, finishing, etc.) can be considered as one of the properties and given the appropriate weighting factor. However, if there is a large number of properties to consider, the importance of cost may be emphasized by considering it separately as a modifier to the material performance index (γ). In the cases where the material is used for space filling, cost can be introduced on per unit volume basis. A figure of merit (M) for the material can then be defined as:

$$M = \gamma/(C\rho) \tag{7}$$

where C = total cost of the material per unit weight (stock, processing, finishing, etc.)
ρ = density of the material.

When an important function of the material is to bear stresses, it may be more appropriate to use the cost of unit strength instead of the cost per unit volume. This is because higher strength will allow less material to be used to bear the load, and the cost of unit strength may be a better representative of the amount of material actually used in making the part. In this case, Eq. 7 is rewritten as:

$$M = \gamma/C' \tag{8}$$

where C' is determined from Table 1 depending on the type of loading.

This argument may also hold in other cases where the material performs an important function such as electrical conductivity or thermal insulation. In these cases the amount of the material, and consequently the cost, are directly affected by the value of the property.

When a large number of materials with a large number of specified properties are being evaluated for selection, the weighted-properties method can involve a large number of tedious and time-consuming calculations. In such cases, the use of a computer would facilitate the selection process. The steps involved in the weighted-properties method can be written in the form of a simple computer program to select materials from a data bank. An interactive program can also include the digital logic method to help in determining the weighting factors.

4 SELECTING THE OPTIMUM SOLUTION

Candidates that have the most promising performance indices can each now be used to develop a detail design. Each detail design will exploit the points of strength of the material, avoid the weak points, and reflect the requirements of the manufacturing processes needed for the material. The different designs are then compared, taking the cost elements into consideration, in order to arrive at the optimum design–material–process combination.[16]

5 CASE STUDY IN MATERIAL SELECTION

The following case study illustrates the procedure for materials selection as described in Sections 2, 3, and 4 and is based on Ref. 16. The objective is to select the least expensive component that satisfies the requirements for a simple structural component for a sailing-boat mast in the form of a hollow cylinder of length 1000 mm, which is subjected to compressive axial forces of 153 kN. Because of space and weight limitations, the outer diameter of the component should not exceed 100 mm, the inner diameter should not be less than 84 mm, and the mass should not exceed 3 kg. The component will be subjected to mechanical impact and spray of water. Assembly to other components requires the presence of relatively small holes.

5.1 Material Performance Requirements

Possible modes of failure and the corresponding material properties that are needed to resist failure for the present component include:

- Catastrophic fracture due to impact loading, especially near assembly holes, is resisted by high fracture toughness of the material. This is a rigid material requirement and will be used for initial screening of materials.
- Plastic yielding is resisted by high yield strength. This is a soft material requirement, but a lower limit will be determined by the limitation on the outer diameter.
- Local and global buckling are resisted by high elastic modulus. This is a soft material requirement, but a lower limit will be determined by the limitation on the outer diameter.
- Internal fiber buckling for fiber-reinforced materials is resisted by high modulus of elasticity of the matrix and high volume fraction of fibers in the loading direction. This is a soft material requirement, but a lower limit will be determined by the limitation on the outer diameter.
- Corrosion, which can be resisted either by selecting materials with inherently good corrosion resistance or by protective coating.
- Reliability of the component in service. A factor of safety of 1.5 is taken for the axial loading, i.e., the working axial force will be taken as 230 kN in order to improve reliability.

In addition to the above requirements the limitations set on dimensions and weight should be observed.

5.2 Initial Screening of Materials

The requirement for fracture toughness of the material is used to eliminate ceramic materials. Because of the limitations set on the outer and inner diameters, the maximum possible cross section of the component is about 2300 mm^2. To avoid yielding under the axial working load, the yield strength of the material should be more than 100 MPa, which excludes engineering polymers, woods, and some of the lower strength engineering alloys; see Fig. 2. Corrosion resistance is desirable but will not be considered a factor for screening since the possibility of protection for less corrosion materials exists but will be considered as a soft requirement.

5.3 Comparing Alternative Solutions

Table 3 shows a sample of materials that satisfy the conditions set in the initial screening stage. In a real-life situation the list in the table could be much longer,

Table 3 Properties of Sample Candidate Materials[16]

Material	Yield Strength (MPa)	Elastic Modulus (GPa)	Specific Gravity	Corrosion Resistance[a]	Cost Category[b]
AISI 1020 (UNS G10200)	280	210	7.8	1	5
AISI 1040 (UNS G10400)	400	210	7.8	1	5
ASTM A242 type 1 (UNS K11510)	330	212	7.8	1	5
AISI 4130 (UNS G41300)	1520	212	7.8	4	3
AISI 316 (UNS S31600)	205	200	7.98	4	3
AISI 416 heat treated (UNS S41600)	440	216	7.7	4	3
AISI 431 heat treated (UNS S43100)	550	216	7.7	4	3
AA 6061 T6 (UNS A96061)	275	69.7	2.7	3	4
AA 2024 T6 (UNS A92024)	393	72.4	2.77	3	4
AA 2014 T6 (UNS A92014)	415	72.1	2.8	3	4
AA 7075 T6 (UNS A97075)	505	72.4	2.8	3	4
Ti–6Al–4V	939	124	4.5	5	1
Epoxy–70% glass fabric	1270	28	2.1	4	2
Epoxy–63% carbon fabric	670	107	1.61	4	1
Epoxy–62% aramid fabric	880	38	1.38	4	1

[a] 5 Excellent, 4 Very good, 3 Good, 2 Fair, 1 Poor.

[b] 5 Very inexpensive, 4 Inexpensive, 3 Moderate price, 2 Expensive, 1 Very expensive.

but the intent here is to illustrate the procedure. The yield strength, elastic modulus, specific gravity, corrosion resistance, and cost category are given for each of the materials. At this stage, it is sufficient to classify materials into very inexpensive, inexpensive, etc. Better estimates of the material and manufacturing cost will be needed in making the final decision in selection. Because the weight of the component is important in this application, specific strength and specific modulus would be better indicators of the suitability of the material (Table 4). The relative importance of the material properties is given in Table 5, and the performance indices of the different materials, as determined by the weighted-properties method, are given in Table 6. The seven candidate materials with high-performance indices ($\gamma > 45$) are selected for making actual component designs.

5.4 Selecting the Optimum Solution

As shown earlier, the possible modes of failure of a hollow cylinder include yielding, local and global buckling, and internal fiber buckling. These four failure modes are used to develop the design formulas for the mast component. For

Table 4 Properties of Sample Candidate Materials[16]

Material	Specific Strength (MPa)	Specific Modulus (GPa)	Corrosion Resistance[a]	Cost Category[b]
AISI 1020 (UNS G10200)	35.9	26.9	1	5
AISI 1040 (UNS G10400)	51.3	26.9	1	5
ASTM A242 type 1 (UNS K11510)	42.3	27.2	1	5
AISI 4130 (UNS G41300)	194.9	27.2	4	3
AISI 316 (UNS S31600)	25.6	25.1	4	3
AISI 416 heat treated (UNS S41600)	57.1	28.1	4	3
AISI 431 heat treated (UNS S43100)	71.4	28.1	4	3
AA 6061 T6 (UNS A96061)	101.9	25.8	3	4
AA 2024 T6 (UNS A92024)	141.9	26.1	3	4
AA 2014 T6 (UNS A92014)	148.2	25.8	3	4
AA 7075 T6 (UNS A97075)	180.4	25.9	3	4
Ti–6Al–4V	208.7	27.6	5	1
Epoxy–70% glass fabric	604.8	28	4	2
Epoxy–63% carbon fabric	416.2	66.5	4	1
Epoxy–62% aramid fabric	637.7	27.5	4	1

[a] 5 Excellent, 4 Very good, 3 Good, 2 Fair, 1 Poor.

[b] 5 Very inexpensive, 4 Inexpensive, 3 Moderate price, 2 Expensive, 1 Very expensive.

more details on the design and optimization procedure or Eqs. 9–12, please refer to Ref. 16.

$$\text{Condition for yielding: } F/A < \sigma_y \tag{9}$$

where σ_y = yield strength of the material
F = external working axial force
A = cross sectional area

$$\text{Condition for local buckling: } F/A < 0.121ES/D \tag{10}$$

Table 5 Weighting Factors

Property	Specific Strength (MPa)	Specific Modulus (GPa)	Corrosion Resistance	Relative Cost
Weighting factor (α)	0.3	0.3	0.15	0.25

Table 6 Calculation of the Performance Index

Material	Scaled Specific Strength * 0.3	Scaled Specific Modulus * 0.3	Scaled Corrosion Resistance * 0.15	Scaled Relative Cost * 0.25	Performance Index (γ)
AISI 1020 (UNS G10200)	1.7	12.3	3	25	42
AISI 1040 (UNS G10400)	2.4	12.3	3	25	42.7
ASTM A242 type 1 (UNS K11510)	2	12.3	3	25	42.3
AISI 4130 (UNS G41300)	9.2	12.3	6	15	42.5
AISI 316 (UNS S31600)	1.2	11.3	12	15	39.5
AISI 416 heat treated (UNS S41600)	2.7	12.7	12	15	42.4
AISI 431 heat treated (UNS S43100)	3.4	12.7	12	15	43.1
AA 6061 T6 (UNS A96061)	4.8	11.6	9	20	45.4
AA 2024 T6 (UNS A92024)	6.7	11.8	9	20	47.5
AA 2014 T6 (UNS A92014)	7	11.6	9	20	47.6
AA 7075 T6 (UNS A97075)	8.5	11.7	9	20	49.2
Ti–6Al–4V	9.8	12.5	15	5	42.3
Epoxy–70% glass fabric	28.4	12.6	12	10	63
Epoxy–63% carbon fabric	19.6	30	12	5	66.6
Epoxy–62% aramid fabric	30	12.4	12	5	59.4

where D = outer diameter of the cylinder
S = wall thickness of the cylinder
E = elastic modulus of the material

Condition for global buckling:

$$\sigma_y > F/A[1 + (LDA/1000I)\sec\{(F/EI)^{1/2}L/2\}] \tag{11}$$

where I = second moment of area
L = length of the component

Condition for internal fiber buckling:

$$F/A < [E_m/4(1 + \nu_m)(1 - V_f^{1/2})] \tag{12}$$

where E_m = elastic modulus of the matrix material
ν_m = Poisson's ratio of the matrix material
V_f = volume fraction of the fibers parallel to the loading direction

Figure 3 shows the optimum design range of component diameter and wall

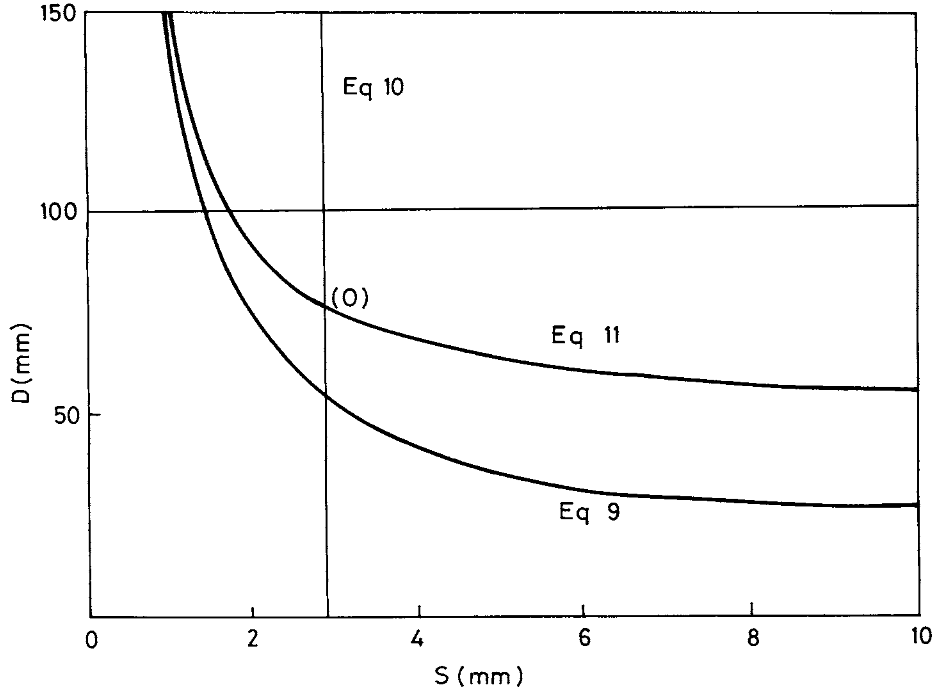


Fig. 3 Design range as predicted by Eqs. 9–11 for AA 7075 aluminum alloy. (Reprinted from *Materials and Design,* **13**, M. M. Farag and E. El-Magd, An Integrated Approach to Product Design, Materials Selection, and Cost Estimation, 323–327, © 1992, with permission from Elsevier Science.)

thickness as predicted by Eqs. 9–11 for AA 7075 aluminum alloy. Point (*O*) represents the optimum design. Similar figures were developed for the different candidate materials to determine the mast component's optimum design dimensions when made of the materials and the results as shown in Table 7. Although all the materials in Table 7 can be used to make safe components that comply

Table 7 Designs Using Candidate Materials with Highest Performance Indices[16]

Material	D_a (mm)	S (mm)	A (mm^2)	Mass (kg)	Cost/kg ($)	Cost of Component ($)
AA 6061 T6 (UNS A96061)	100	3.4	1065.7	2.88	8	23.2
AA 2024 T6 (UNS A92024)	88.3	2.89	801.1	2.22	8.3	18.4
AA 2014 T6 (UNS A92014)	85.6	2.89	776.6	2.17	9	19.6
AA 7075 T6 (UNS A97075)	78.1	2.89	709.1	1.99	10.1	20
Epoxy–70% glass fabric	78	4.64	1136.3	2.39	30.8	73.6
Epoxy–63% carbon fabric	73.4	2.37	546.1	0.88	99	87.1
Epoxy–62% aramid fabric	75.1	3.99	941.6	1.30	88	114.4

with the space and weight limitations, AA 2024 T6 is selected since it gives the least expensive solution.

6 MATERIALS SUBSTITUTION

The common reasons for materials substitution include:

- Taking advantage of new materials or processes
- Improving service performance, including longer life and higher reliability
- Meeting new legal requirements
- Accounting for changed operating conditions
- Reducing cost and making the product more competitive

Generally, a simple substitution of one material for another does not produce an optimum solution. This is because it is not possible to realize the full potential of a new material unless the component is redesigned to exploit its strong points and manufacturing characteristics. Following is a brief description of some of the quantitative methods that are available for making decisions in materials substitution.

6.1 Pugh Method

The Pugh method[17] is useful as an initial screening method in the early stages of design. In this method, a decision matrix is constructed as shown in Table 8. Each of the properties of a possible alternative new material is compared with the corresponding property of the currently used material and the result is recorded in the decision matrix as (+) if more favorable, (−) if less favorable, and (0) if the same. The decision on whether a new material is better than the currently used material is based on the analysis of the result of comparison, i.e., the total number of (+), (−), and (0). New materials with more favorable properties than drawbacks are selected as serious candidates for substitution and are used to redesign the component and for detailed analysis.

Table 8 Example of Use of Pugh Decision Matrix for Materials Substitution

Property	Currently Used Material	New Material (1)	New Material (2)	New Material (3)
Property 1	C1	−	+	+
Property 2	C2	+	+	+
Property 3	C3	+	+	−
Property 4	C4	0	+	−
Property 5	C5	−	0	−
Property 6	C6	0	0	0
Property 7	C7	−	−	0
Property 8	C8	−	+	0
Property 9	C9	−	0	0
Total (+)		2	5	2
Total (−)		5	1	3
Total (0)		2	3	4

6.2 Cost–Benefit Analysis

The cost–benefit analysis is more suitable for the detailed analysis involved in making the final material substitution decision.[1] Because new materials are usually more complex and often require closer control and even new technologies for their processing, components made from such materials are more expensive. This means that for materials substitution to be economically feasible, the economic gain as a result of improved performance ΔB should be more than the additional cost incurred as a result of substitution ΔC.

$$\Delta B - \Delta C > 1 \tag{13}$$

For this analysis it is convenient to divide the cost of materials substitution ΔC into:

- *Cost Differences in Direct Material and Labor.* New materials often have better performance but are more expensive. When smaller amounts of the new material are used to make the product, the increase in direct material cost may not be as great as it would appear at first. Cost of labor may not be an important factor in substitution if the new materials do not require new processing techniques and assembly procedures. If, however, new processes are needed, new cycle times may result and the difference in productivity has to be carefully assessed.
- *Cost of Redesign and Testing.* Using new materials usually involves design changes and testing of components to ensure that their performance meets the requirements. The cost of redesign and testing can be considerable in the case of critical components.
- *Cost of New Tools and Equipment.* Changing materials can have considerable effect on life and cost of tools, and it may influence the heat treatment and finishing processes. This can be a source of cost saving if the new material does not require the same complex treatment or finishing processes used for the original material. The cost of equipment needed to process new materials can be considerable if the new materials require new production facilities as in the case of replacing metals with plastics.

Based on the above analysis, the total cost (ΔC) of substituting a new material, n, in place of an original material, o, in a given part is:

$$\Delta C = (P_n M_n - P_o M_o) + f(C_t/N) + (T_n - T_o) + (L_n - L_o) \tag{14}$$

where P_n, P_o = price/unit mass of new and original materials used in the part
M_n, M_o = mass of new and original materials used in the part
f = capital recovery factor; it can be taken as 15% in the absence of information
C_t = cost of transition from original to new materials
N = total number of new parts produced
T_n, T_o = tooling cost per part for new and original materials
L_n, L_o = labor cost per part using new and old materials

The gains as a result of improved performance ΔB can be estimated based on the expected improved performance of the component, which can be related to the increase in performance index of the new material compared with the currently used material. Such increases include the saving gained as a result of weight reduction or increased service life of the component.

$$\Delta B = A(\gamma_n - \gamma_o) \tag{15}$$

where γ_n, γ_o = performance indices of the new and original materials, respectively

A = benefit of improved performance of the component expressed in dollars per unit increase in material performance index γ.

7 CASE STUDY IN MATERIALS SUBSTITUTION

In the case study in materials selection that was discussed in Section 5, the aluminum alloy AA 2024 T6 was selected since it gives the least expensive solution. Of the seven materials in Table 7, AA 6061 T6, epoxy–70% glass fabric, and epoxy–62% aramid fabric result in components that are heavier and more expensive than those of the other four materials and will be rejected as they offer no advantage. Of the remaining four materials, AA 2024 T6 results in the least expensive but the heaviest component. The other three materials—AA 2014 T6, AA 7075 T6, and epoxy–63% carbon fabric—result in progressively lighter components at progressively higher cost.

For the cases where it is advantageous to have a lighter component, the cost–benefit analysis can be used in finding a suitable substitute for AA 2024 T6 alloy. For this purpose Eq. 15 is used with the performance index γ being considered as the weight of the component and ΔC being the difference in cost of component and A is the benefit expressed in dollars, of reducing the mass by 1 kg. Comparing the materials in pairs shows that:

For $A < \$7/\text{kg}$ saved,	AA 2024 T6 is the optimum material.
For $A = \$7 - \$60.5/\text{kg}$ saved,	AA 7075 T6 is a better substitute.
For $A > \$60.5/\text{kg}$ saved,	Epoxy–63% carbon fabric is optimum.

8 SOURCES OF INFORMATION AND COMPUTER-ASSISTED SELECTION

One essential requisite to successful materials selection is a source of reliable and consistent data on materials properties. There are many sources of information, which include governmental agencies, trade associations, engineering societies, textbooks, research institutes, and materials producers. The ASM International has recently published a directory of materials property databases[18] that contains more than 500 data sources, including both specific databases and data centers. For each source, the directory gives a brief description of the available information, address, telephone number, e-mail, web site, and approximate cost if applicable. The directory also has indices by material and by property to help the user in locating the most appropriate source of material information. Much of the information is available on CD-ROM or PC disk, which makes it possible to integrate the data source in computer-assisted selection systems.

Other useful reviews of the sources of materials property data and information are also given in Refs. 19 and 20.

8.1 Computerized Materials Databases

Computerized materials databases are an important part of any computer-aided system for selection. With an interactive database, as in the case of *ASM Metal Selector,*[21] the user can define and redefine the selection criteria to gradually sift the materials and isolate the candidates that meet the requirements. In many cases, sifting can be carried out according to different criteria such as:

1. Specified numeric values of a set of material properties
2. Specified level of processability such as machinability, weldability, formability, availability, processing cost, etc.
3. Class of material, e.g., fatigue resistant, corrosion resistant, heat resistant, electrical materials, etc.
4. Forms such as rod, wire, sheet, tube, cast, forged, welded, etc.
5. Designations: Unified Numbering System (UNS) numbers, American Iron and Steel Institute (AISI) numbers, common names, material group or country of origin
6. Specifications, which allows the operator to select the materials that are acceptable to organizations such as the American Society for Testing and Materials (ASTM) and the Society of Automotive Engineers (SAE)
7. Composition, which allows the operator to select the materials that have certain minimum and/or maximum values of alloying elements

More than one of the above sifting criteria can be used to identify suitable materials. Sifting can be performed in the AND or OR modes. The AND mode narrows the search since the material has to conform to all the specified criteria. The OR mode broadens the search since materials that satisfy any of the requirements are selected.

The number of materials that survive the sifting process depends on the severity of the criteria used. At the start of sifting, the number of materials shown on the screen is the total in the database. As more restrictions are placed on the materials, the number of surviving materials gets smaller and could reach 0, i.e., no materials qualify. In such cases, some of the restrictions have to be relaxed and the sifting restarted.

8.2 Computer Assistance in Making Final Selection

Integrating material property database with design algorithms and computer-aided design (CAD)/computer-aided manufacturing (CAM) programs has many benefits including homogenization and sharing of data in the different departments, decreased redundancy of effort, and decreased cost of information storage and retrieval. Several such systems have been cited in Ref. 18, including:

- The Computerized Application and Reference System (CARS), developed from the AISI *Automotive Steel Design Manual*, performs first-order analysis of design using different steels.

- Aluminum Design System (ADS), developed by the Aluminum Association (U.S.), performs design calculations and conformance checks of aluminum structural members with the design specifications for aluminum and its alloys.
- Material Selection and Design for fatigue life predictions, developed by ASM International, aids in the design of machinery and engineering structures using different engineering materials.
- Machine Design's Materials Selection, developed by Penton Media (U.S.), combines the properties for a wide range of materials and the data set for design analysis.

8.3 Expert Systems

Expert systems, also called knowledge-based systems, are computer programs that simulate the reasoning of a human expert in a given field of knowledge. Expert systems rely on heuristics, or rules of thumb, to extract information from a large knowledge base. Expert systems typically consist of three main components:

- The knowledge base contains facts and expert-level heuristic rules for solving problems in a given domain. The rules are normally introduced to the system by domain experts through a knowledge engineer.
- The inference engine provides an organized procedure for sifting through the knowledge base and choosing applicable rules in order to reach the recommend solutions. The inference engine also provides a link between the knowledge base and the user interface.
- The user interface allows the user to input the main parameters of the problem under consideration. It also provides recommendations and explanations of how such recommendations were reached.

A commonly used format for the rules in the knowledge base is in the form:

IF (condition 1) and/or (condition 2)
THEN (conclusion 1)

For example, in the case of FRP selection:

IF: required elastic modulus, expressed in GPa, is more than 150 and specific gravity less than 1.7.
THEN: oriented carbon fibers at 60% by volume.

Expert systems are finding many applications in industry including the areas of design, trouble-shooting, failure analysis, manufacturing, materials selection, and materials substitution.[12] When used to assist in materials selection, expert systems provide impartial recommendations and are able to search large databases for optimum solutions. Another important advantage of expert systems is their ability to capture valuable expertise and make it available to a wider circle of users. An example is the Chemical Corrosion Expert System, which is produced by the National Association of Corrosion Engineers (NACE) in the United States.[18] The system prompts the user for information on the environmental conditions and configuration of the component and then recommends candidate materials.

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PART 2

MAJOR MATERIALS

Handbook of Materials Selection. Edited by Myer Kutz

CHAPTER 2

CARBON AND ALLOY STEELS

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1 INTRODUCTION

Steel is the most common and widely used metallic material in today's society. It can be cast or wrought into numerous forms and can be produced with tensile strengths exceeding 5 GPa. A prime example of the versatility of steel is in the automobile where it is the material of choice and accounts for over 60% of the weight of the vehicle. Steel is highly formable as seen in the contours of the automobile outerbody. Steel is strong and is used in the body frame, motor brackets, driveshaft, and door impact beams of the vehicle. Steel is corrosion resistant when coated with the various zinc-based coatings available today. Steel is dent resistant when compared with other materials and provides exceptional energy absorption in a vehicle collision. Steel is recycled and easily separated from other materials by a magnet. Steel is inexpensive compared with other competing materials such as aluminum and various polymeric materials.

In the past, steel has been described as an alloy of iron and carbon. Today, this description is no longer applicable since in some very important steels, e.g., interstitial-free (IF) steels and type 409 ferritic stainless steels, carbon is considered an impurity and is present in quantities of only a few parts per million. By definition, steel must be at least 50% iron and must contain one or more alloying element. These elements generally include carbon, manganese, silicon, nickel, chromium, molybdenum, vanadium, titanium, niobium, and aluminum.

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Each chemical element has a specific role to play in the steelmaking process or in achieving particular properties or characteristics, e.g., strength, hardness, corrosion resistance, magnetic permeability, and machinability.

2 STEEL MANUFACTURE

In most of the world, steel is manufactured by integrated steel facilities that produce steel from basic raw materials, i.e., iron ore, coke, and limestone. However, the fastest growing segment of the steel industry is the "minimill" that melts steel scrap as the raw material. Both types of facilities produce a wide variety of steel forms including sheet, plate, structural, railroad rail, and bar products.

Ironmaking. When making steel from iron ore, a blast furnace chemically reduces the ore (iron oxide) with carbon in the form of coke. Coke is a spongelike carbon mass that is produced from coal by heating the coal to expel the organic matter and gasses. Limestone (calcium carbonate) is added as a flux for easier melting and slag formation. The slag, which floats atop the molten iron, absorbs many of the unwanted impurities. The blast furnace is essentially a tall hollow cylindrical structure with a steel outer shell lined on the inside with special refractory and graphite brick. The crushed or pelletized ore, coke, and limestone are added as layers through an opening at the top of the furnace, and chemical reduction takes place with the aid of a blast of preheated air entering near the bottom of furnace (an area called the bosh). The air is blown into the furnace through a number of water-cooled copper nozzles called tuyeres. The reduced liquid iron fills the bottom of the furnace and is tapped from the furnace at specified intervals of time. The product of the furnace is called pig iron because in the early days the molten iron was drawn from the furnace and cast directly into branched mold configurations on the cast house floor. The central branch of iron leading from the furnace was called the "sow" and the side branches were called "pigs." Today the vast majority of pig iron is poured directly from the furnace into a refractory-lined vessel (submarine car) and transported in liquid form to a basic oxygen furnace (BOF) for refinement into steel.

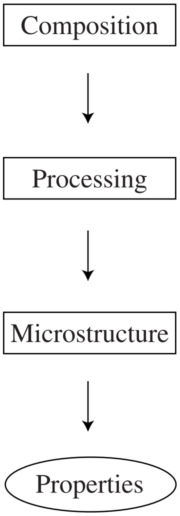
Steelmaking. In the BOF, liquid pig iron comprises the main charge. Steel scrap is added to dilute the carbon and other impurities in the pig iron. Oxygen gas is blown into the vessel by means of a top lance submerged below the liquid surface. The oxygen interacts with the molten pig iron to oxidize undesirable elements. These elements include excess carbon (because of the coke used in the blast furnace, pig iron contains over 2% carbon), manganese, and silicon from the ore and limestone and other impurities like sulfur and phosphorus. While in the BOF, the liquid metal is chemically analyzed to determine the level of carbon and impurity removal. When ready, the BOF is tilted and the liquid steel is poured into a refractory-lined ladle. While in the ladle, certain alloying elements can be added to the steel to produce the desired chemical composition. This process takes place in a ladle treatment station or ladle furnace where the steel is maintained at a particular temperature by external heat from electrodes in the lid placed on the ladle. After the desired chemical composition is achieved, the ladle can be placed in a vacuum chamber to remove undesirable gases such as hydrogen and oxygen. This process is called degassing and is used for higher quality steel products such as railroad rail, sheet, plate, bar, and forged products. Stainless steel grades are usually produced in an induction or electric arc furnace, sometimes under vacuum. To refine stainless steel, the argon–oxygen decarburization (AOD) process is used. In the AOD, an argon–oxygen gas mixture is injected through the molten steel to remove carbon without a substantial loss of chromium (the main element in stainless steel).

Continuous Casting. Today, most steel is cast into solid form in a continuous-casting (also called strand casting) machine. Here, the liquid begins solidification in a water-cooled copper mold while the steel billet, slab, or bloom is withdrawn from the bottom of the mold. The partially solidified shape is continuously withdrawn from the machine and cut to length for further processing. The continuous-casting process can proceed for days or weeks as ladle after ladle of molten steel feeds the casting machine. Some steels are not continuously cast but are poured into individual cast-iron molds to form an ingot that is later reduced in size by forging or a rolling process to some other shape. Since the continuous-casting process offers substantial economic and quality advantages over ingot casting most steel in the world is produced by continuous casting.

Rolling/Forging. Once cast into billet, slab, or bloom form, the steel is hot rolled through a series of rolling mills or squeezed/hammered by forging to produce the final shape. To form hot-rolled sheet, a 50- to 300-mm-thick slab is reduced to final thickness, e.g., 2 mm, in one or more roughing stands followed by a series of six or seven finishing stands. To obtain thinner steel sheet, e.g., 0.5 mm, the hot-rolled sheet must be pickled in acid to remove the iron oxide scale and further cold rolled in a series of rolling stands called a tandem mill. Because the cold-rolling process produces a hard sheet with little ductility, it is annealed either by batch annealing or continuous annealing. New casting technology is emerging where thin sheet (under 1 mm) can be directly cast from the liquid through water-cooled, rotating rolls that act as a mold as in continuous casting. This new process eliminates many of the steps in conventional hot-rolled sheet processing. Plate steels are produced by hot rolling a slab in a reversing roughing mill and a reversing finishing mill. Steel for railway rails is hot rolled from a bloom in a blooming mill, a roughing mill, and one or more finishing mills. Steel bars are produced from a heated billet that is hot rolled in a series of roughing and finishing mills. Forged steels are produced from an ingot that is heated to forging temperature and squeezed or hammered in a hydraulic press or drop forge. The processing sequence in all these deformation processes can vary depending on the design, layout, and age of the steel plant.

3 DEVELOPMENT OF STEEL PROPERTIES

In order to produce a steel product with the desired properties, basic metallurgical principles are used to control three things,



This means that the steel composition and processing route must be closely controlled in order to produce the proper microstructure. The final microstructure is of utmost importance in determining the properties of the steel product. This section will explore how various microstructures are developed and the unique characteristics of each microstructural component in steel. The next section will discuss how alloy composition also plays a major role.

Iron–Carbon Equilibrium Diagram. Since most steels contain carbon, the basic principles of microstructural development can be explained by the iron–carbon equilibrium diagram. This diagram, shown in Fig. 1, is essentially a map of the phases that exist in iron at various carbon contents and temperatures under equilibrium conditions. Iron is an interesting chemical element in that it undergoes three phase changes when heated from room temperature to liquid. For example, from room temperature to 912°C, pure iron exists as ferrite (also called alpha iron), from 912 to 1394°C, it exists as austenite (gamma iron), from 1394 to 1538°C it exists as ferrite again (delta iron), and above 1538°C it is liquid. In other words, upon heating, iron undergoes allotropic phase transformations from ferrite to austenite at 912°C, austenite to ferrite at 1394°C, and ferrite to liquid at 1538°C. Each transformation undergoes a change in crystal structure or arrangement of the iron atoms in the crystal lattice. It must be remembered that all chemical elements in their solid form have specific arrangements of atoms that are essentially the basic building blocks in producing the element in the form that we physically observe. These atomic arrangements form a latticework containing billions of atoms all aligned in a systematic way. Some of these lattices have a cubic arrangement, with an atom at each corner of the cube and another atom at the cube center. This arrangement is called body-centered-cubic (bcc). Others have an atom at each corner of the cube and atoms at the center of each face of the cube. This is called face-centered-cubic (fcc). Other arrangements are hexagonal, some are tetragonal, etc. As an example, pure iron as ferrite has a bcc arrangement. Austenite has a fcc arrangement. Upon heating, bcc

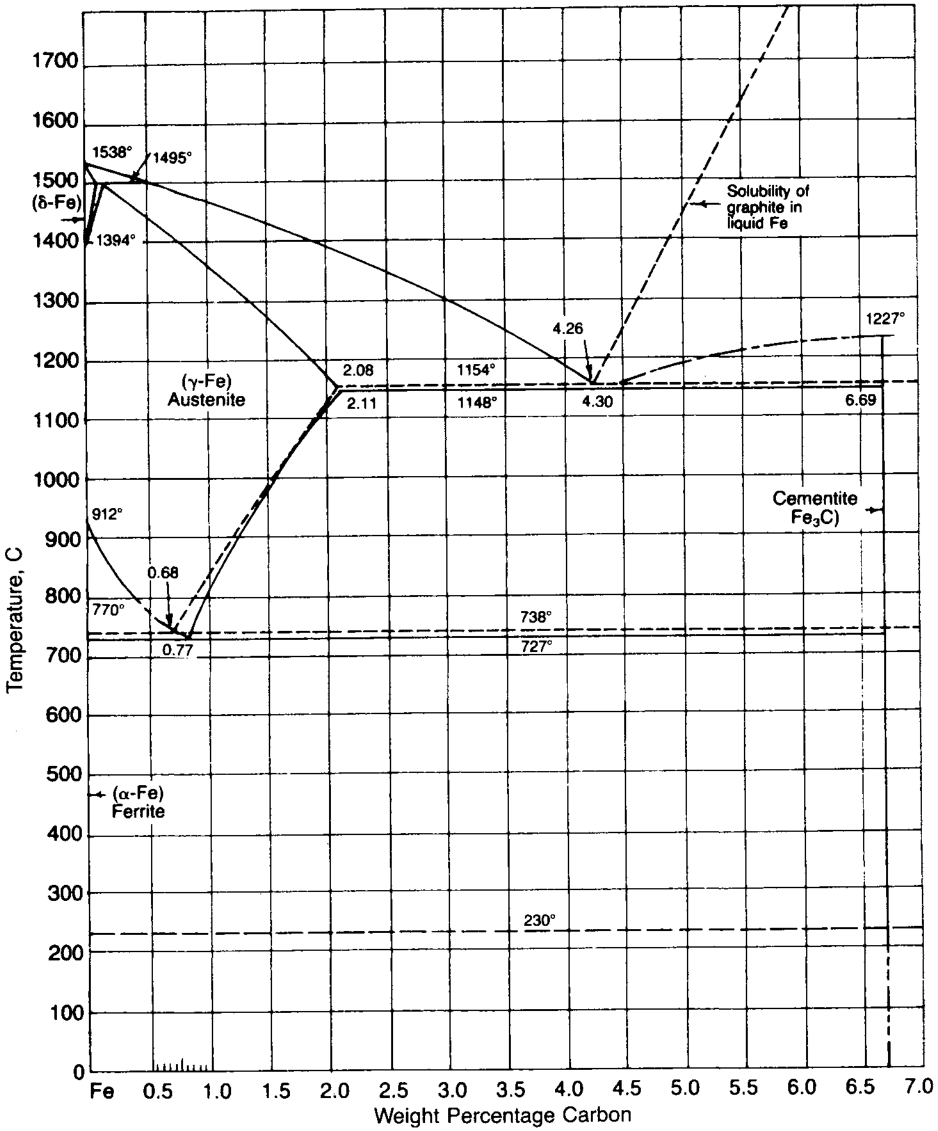


Fig. 1 Iron–carbon binary-phase diagram. (*Source: Steels: Heat Treatment and Processing Principles,* ASM International, Materials Park, OH 44073-0002, 1990, p. 2.)

ferrite will transform to fcc austenite at 912°C. These arrangements or crystal structures impart different properties to steel. For example, a bcc ferritic stainless steel will have properties much different from a fcc austenitic stainless steel as described later in this chapter.

Since pure iron is very soft and of low strength, it is of little interest commercially. Therefore, carbon and other alloying elements are added to enhance properties. Adding carbon to pure iron has a profound effect on ferrite and austenite discussed above. One way to understand the effect of carbon is to examine the iron–carbon diagram (Fig. 1). This is a binary (two-element) diagram of temperature and composition (carbon content) constructed under near-equilibrium conditions. In this diagram, as carbon is added to iron, the ferrite and austenite phase fields expand and contract depending upon the carbon level and temperature. Also, there are fields consisting of two phases, e.g., ferrite plus austenite.

Since carbon has a small atomic diameter when compared with iron, it is called an interstitial element because it can fill the interstices between the iron atoms in the cubic lattice. Nitrogen is another interstitial element. On the other hand, elements such as manganese, silicon, nickel, chromium, and molybdenum have atomic diameters similar to iron and are called substitutional alloying elements. These substitutional elements can thus replace iron atoms at the cube corners, faces, or center positions. There are many binary phase diagrams (Fe–Mn, Fe–Cr, Fe–Mo, etc.) and tertiary-phase diagrams (Fe–C–Mn, Fe–C–Cr, etc.) showing the effect of interstitial and substitutional elements on the phase fields of ferrite and austenite. These diagrams are found in the handbooks listed at the end of the chapter.

Being an interstitial or a substitutional element is important in the development of steel properties. Interstitial elements such as carbon can move easily about the crystal lattice whereas a substitutional element such as manganese is much more difficult to move. The movement of elements in a crystal lattice is called diffusion. Diffusion is a controlling factor in the development of microstructure. Another factor is solubility, which is a measure of how much of a particular element can be accommodated by the crystal lattice before it is rejected. In metals when two or more elements are soluble in the crystal lattice, a solid solution is created (somewhat analogous to a liquid solution of sugar in hot coffee). For example, when added to iron, carbon has very limited solubility in ferrite but is about 100 times more soluble in austenite, as seen in the iron–carbon diagram in Fig. 2 (a limited version of the diagram in Fig. 1). The maximum solubility of carbon in ferrite is about 0.022% C at 727°C while the maximum solubility of carbon in austenite is 100 times more, 2.11% C at 1148°C. At room temperature the solubility of carbon in iron is only about 0.005%. Any amount of carbon in excess of the solubility limit is rejected from solid solution and is usually combined with iron to form an iron carbide compound called cementite. This hard and brittle compound has the chemical formula Fe_3C and a carbon content of 6.7%. This is illustrated in the following two examples. The first example is a microstructure of a very low carbon steel (0.002% C) is shown in Fig. 3*a*. The microstructure consists of only ferrite grains (crystals) and grain boundaries. The second example is a microstructure of a low-carbon steel containing 0.02% C in Fig. 3*b*. In this microstructure, cementite can be seen as particles at the ferrite grain boundaries. The excess carbon rejected from the solid solution of ferrite formed this cementite. As the carbon content in steel is increased, another form of cementite appears as a constituent called pearlite, which can be found in most carbon steels. Examples of pearlite in low-carbon (0.08% C) and medium-carbon (0.20% C) steels are seen in Figs. 4*a* and 4*b*. Pearlite has a lamellar (parallel plates) microstructure as shown at higher magnification in Fig. 5 and consists of layers of ferrite and cementite. Thus, in these examples, in increasing the carbon level from 0.002 to 0.02 to

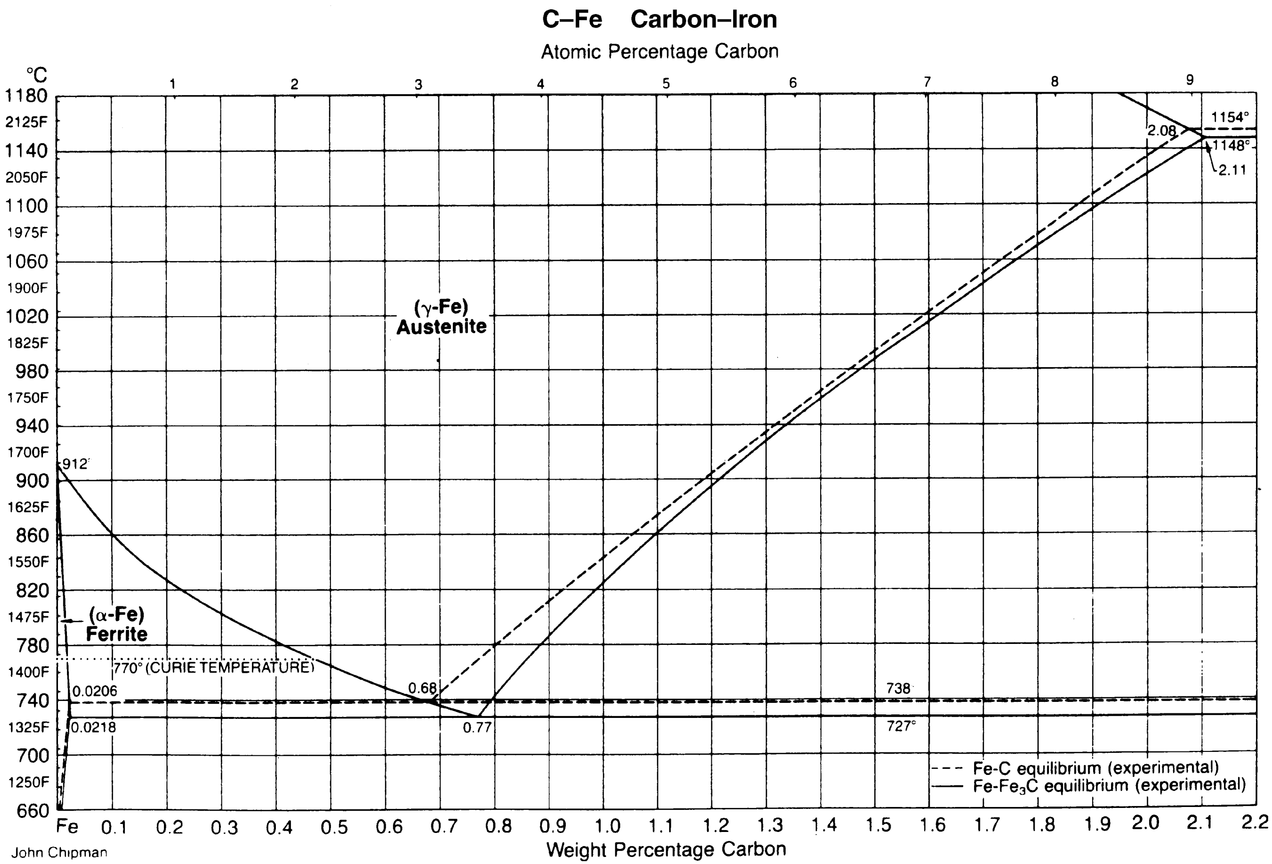
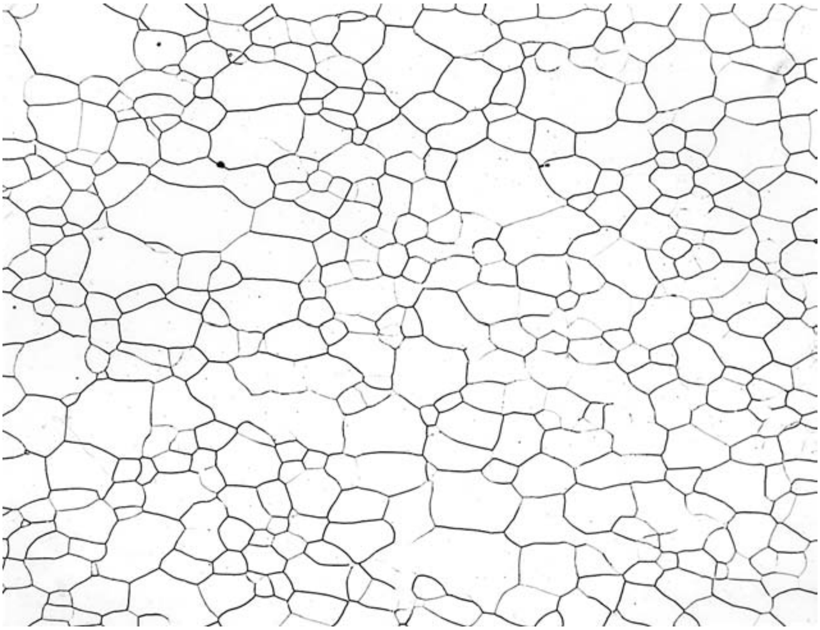
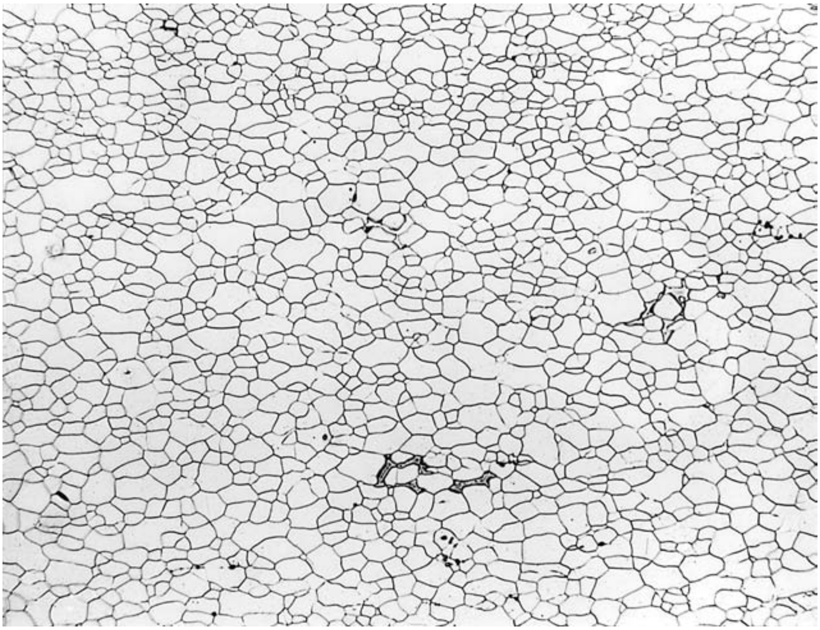


Fig. 2 Expanded portion of the iron–carbon binary-phase diagram in Fig. 1.
(*Source: Steels: Heat Treatment and Processing Principles,* ASM International, Materials Park, OH 44073-0002, 1990, p. 18.)

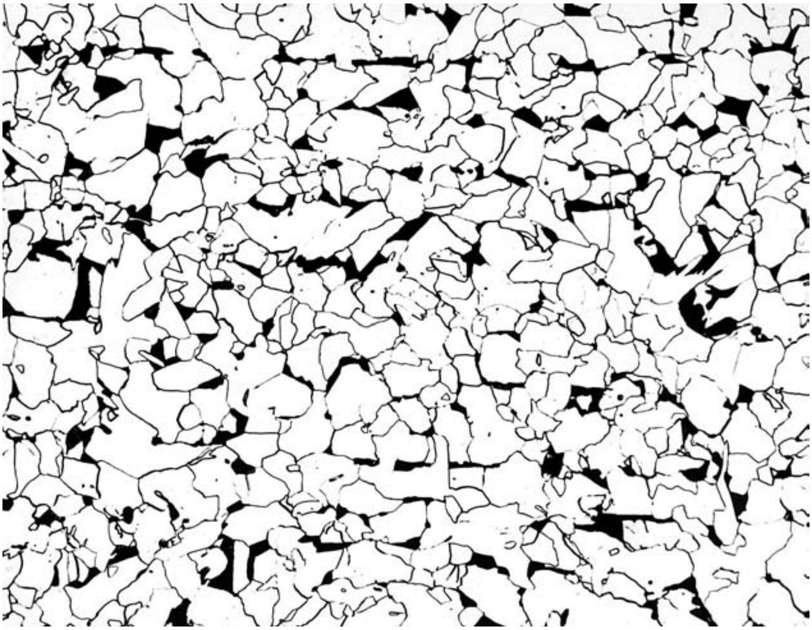


(*a*)



(*b*)

Fig. 3 (*a*) Photomicrograph of a very low carbon steel showing ferrite grains and (*b*) photomicrograph of a low-carbon steel showing ferrite grains with some cementite on the ferrite grain boundaries. (*a*) 500X and (*b*) at 200X. Marshalls etch.



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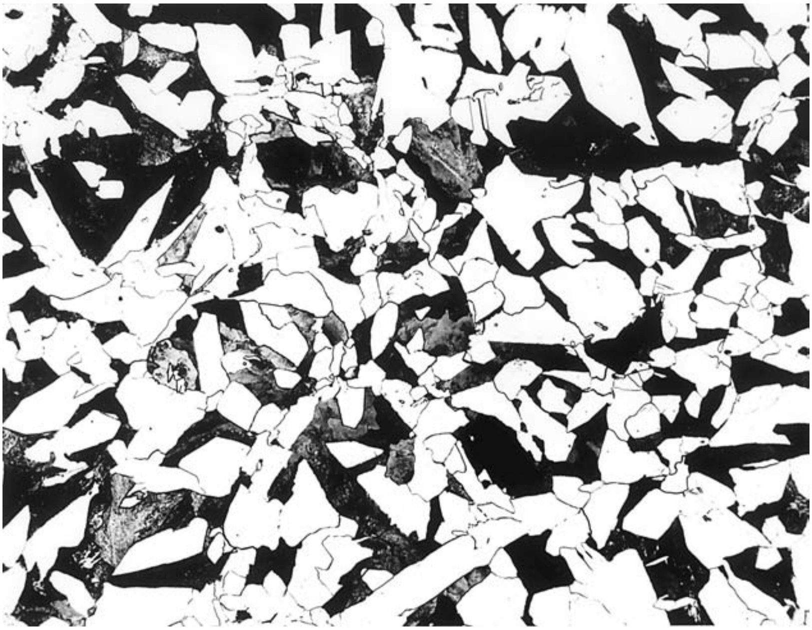


(*b*)

Fig. 4 (*a*) Photomicrograph of an SAE/AISI 1008 steel showing ferrite grains and pearlite (dark) and (*b*) photomicrograph of an SAE/AISI 1020 steel showing ferrite grains with an increased amount of pearlite. (*a*) and (*b*) at 200X. 4% picral + 2% nital etch.

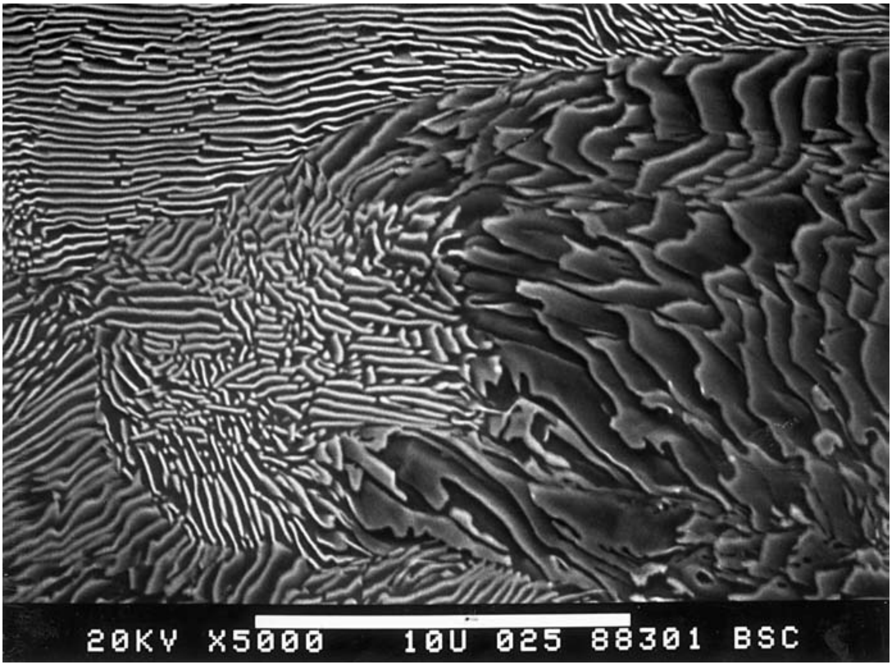


Fig. 5 Scanning electron microscope micrograph of pearlite showing the platelike morphology of the cementite. 5000X. 4% picral etch.

0.08 to 0.20%, the excess carbon is manifested as a carbide phase in two different forms, cementite particles and cementite in pearlite. Both forms increase the hardness and strength of iron. However, there is a trade-off, cementite also decreases ductility and toughness.

Pearlite forms on cooling austenite through a eutectoid reaction as seen below:

$$\text{Austenite} \leftrightarrow \text{Fe}_3\text{C} + \text{Ferrite}$$

A *eutectoid* reaction occurs when a solid phase or constituent reacts to form two different solid constituents on cooling (a *eutectic* reaction occurs when a liquid phase reacts to form two solid phases). The eutectoid reaction is reversible on heating. In steel, the eutectoid reaction (under equilibrium conditions) takes place at 727°C and can be seen on the iron–carbon diagram (Fig. 1) as the "V" at the bottom left side of the diagram. A fully pearlitic microstructure forms at 0.77% C at the eutectoid temperature of 727°C (the horizontal line on the left side of the iron–carbon diagram). Steels with less than 0.77% C are called *hypoeutectoid* steels and consist of mixtures of ferrite and pearlite with the amount of pearlite increasing as the carbon content increases. The ferrite phase is called a *proeutctoid* phase because it forms prior to the eutectoid transformation that occurs at 727°C. A typical example of proeutectoid ferrite is shown in Fig. 6. In this photomicrograph, the ferrite (the white appearing constitu-

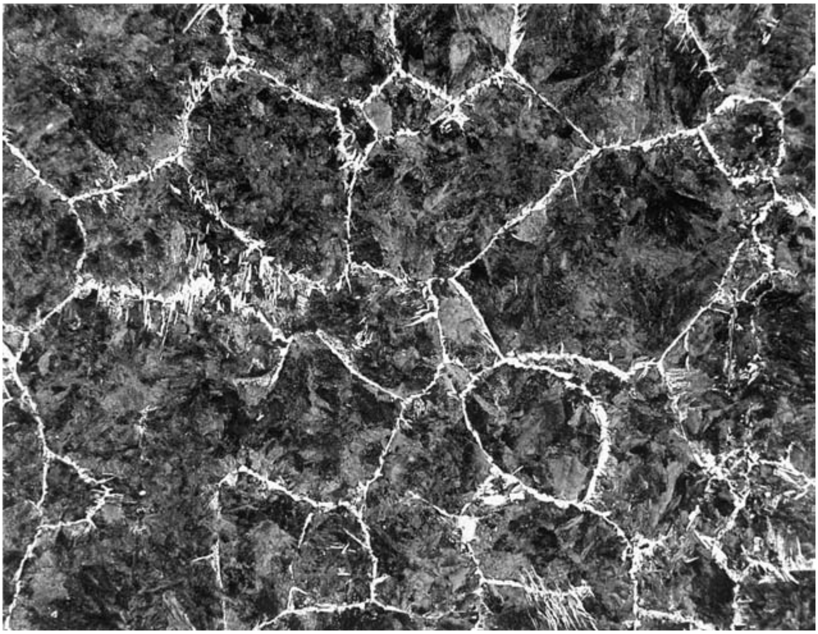


Fig. 6 Photomicrograph of a medium-carbon hypoeutectoid steel showing a pearlite matrix and proeutectoid ferrite nucleating on the original (prior) austenite grain boundaries. 200X. 4% picral + 2% nital etch.

ent) formed on the prior austenite grain boundaries of hypoeutectoid steel with 0.60% C. The remaining constituent (dark appearing) is pearlite. Steels between 0.77% C and about 2% C are called *hypereutectoid* steels and consist of pearlite with proeutectoid cementite. Cementite forms a continuous carbide network at the boundaries of the prior austentite grains. Because there is a carbide network, hypereutectoid steels are characterized as steels with little or no ductility and very poor toughness. This means that in the commercial world the vast majority of carbon steels are hypoeutectoid steels.

Thus, according to the iron–carbon diagram, steels that are processed under equilibrium or near-equilibrium conditions can form (a) pure ferrite at very low carbon levels generally under 0.005% C, (b) ferrite plus cementite particles at slightly higher carbon levels between 0.005% C and 0.022% C, (c) ferrite plus pearlite mixtures between 0.022% C and 0.77% C, (d) 100% pearlite at 0.77% C, and (e) mixtures of pearlite plus cementite networks between 0.77% C and 2% C. The higher the percentage of cementite, the higher the hardness and strength and lower the ductility and toughness of the steel.

Departure from Equilibrium (Real World). Industrial processes do not occur at equilibrium, and only those processes that take place at extremely slow heating and cooling rates can be considered near equilibrium, and these processes are quite rare. Therefore, under real conditions, the iron–carbon diagram can only be used as a rough guideline since the equilibrium transformation temperatures shift to lower temperatures on cooling and to higher temperatures on heating. If steels are cooled at very fast rates, e.g., quenching in water, the iron–carbon diagram can no longer be used since there is a major departure from equilibrium. In fact, during the quenching of steel, new constituents form that are not associated with the iron–carbon diagram. Therefore, at fast cooling rates the concept of time–temperature transformation (TTT) diagrams must be considered. These diagrams are constructed under isothermal (constant) temperature (called IT diagrams) or continuous-cooling conditions (called CT diagrams). It is important to know how these diagrams are constructed so that we can understand the development of nonequilibrium microstructures, which are so important in carbon and alloy steels.

Isothermal Transformation Diagram. This diagram is formed by quenching very thin specimens of steel in salt baths set at various temperatures. For example, thin specimens of 0.79% C steel can be quenched into seven different liquid salt baths set at 650, 600, 550, 500, 450, 400, and 200°C. The specimens are held for various times at each temperature then pulled from the bath and quickly quenched in cold water. The result will be a diagram called an isothermal transformation (IT) diagram, as shown in Fig. 7. The diagram is essentially a map showing where various constituents form. For example, at 650°C, austenite (A) begins to transform to pearlite if held in the bath for 10 s. The curve drawn through this point is the pearlite transformation start temperature and is labeled beginning of transformation in Fig. 7. At about 100 s the pearlite transformation is finished. The second curve represents the pearlite transformation finish temperature and is labeled the end of transformation in Fig. 7. In this steel, pearlite forms at all temperatures along the start of transformation curve from 727°C (the equilibrium temperature of the iron–carbon diagram) to 540°C, the "nose" of the curve. At the higher transformation temperatures, the pearlite interlamellar spacing (the spacing between cementite plates) is very coarse and decreases in spacing as the temperature is decreased, i.e., nose of the IT diagram is approached. This is an important concept since a steel with a coarse pearlite interlamellar spacing is softer and of lower strength than a steel with a fine pearlite interlamellar spacing. Commercially, rail steels are produced with a pearlitic microstructure, and it has been found that the finer the interlamellar spacing the harder the rail and the better the wear resistance. This means that rails will last longer in track if produced with the finest spacing allowable. Most rail producers employ an accelerated cooling process called head hardening to obtain the necessary conditions to achieve the finest pearlite spacing in the rail head (the point of wheel contact).

If the specimens are quenched to 450°C and held for various times, pearlite does not form. In fact, pearlite does not isothermally transform at transformation temperatures (in this case, salt pot temperatures) below the nose of the diagram in Fig. 7. The new constituent is called bainite, which consists of ferrite laths with small cementite particles (also called precipitates). An example of the microstructure of bainite is shown in Fig. 8. This form of bainite is called upper bainite because it is formed in the upper portion below the nose of the IT diagram (between about 540 and 400°C). Lower bainite, a finer ferrite–carbide

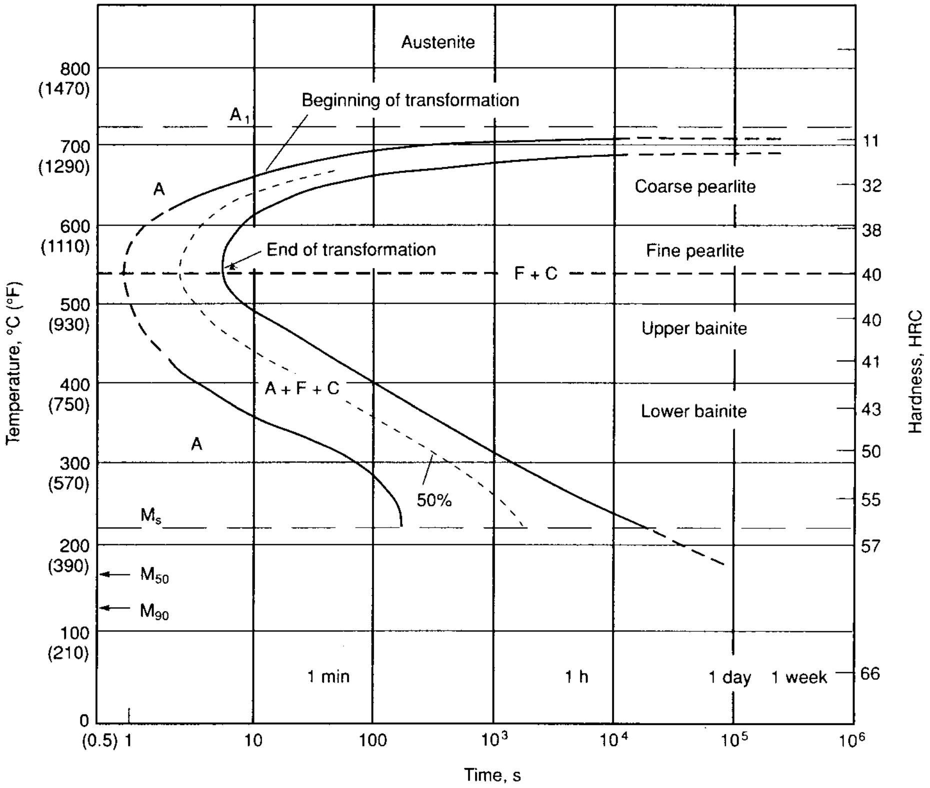


Fig. 7 Isothermal transformation (IT) diagram of SAE/AISI 1080 steel showing the beginning and end of transformation curves with temperature and time. (*Source: ASM Handbook, Vol. 1, Properties and Selection: Irons, Steels, and High-Performance Alloys,* ASM International, Materials Park, OH 44073-0002, 1990, p. 128.)

microstructure, forms at lower temperatures (between 400 and about 250°C). Bainite is an important constituent is tough, high-strength, low-alloy steel.

If specimens are quenched into a salt bath at 200°C, a new constituent called martensite will form. The start of the martensitic transformation is shown in Fig. 7 as M_s (at 220°C). Martensite is a form of ferrite that is supersaturated with carbon. In other words, because of the very fast cooling rate, the carbon atoms do not have time to diffuse from their interstitial positions in the bcc lattice to form cementite particles. An example of martensite is shown in Fig. 9. Steel products produced with an as-quenched martensitic microstructure are very hard and brittle, e.g., a razor blade. Most martensitic products are tempered by heating to temperatures between about 350 and 650°C. The tempering process allows some of the carbon to diffuse and form as a carbide phase from the supersaturated iron lattice. This softens the steel and provides some ductility. The degree of softening is determined by the tempering temperature and the time at the tempering temperature. The higher the temperature and the longer the time the softer the steel. Most steels with martensite are used in the quenched and tempered condition.

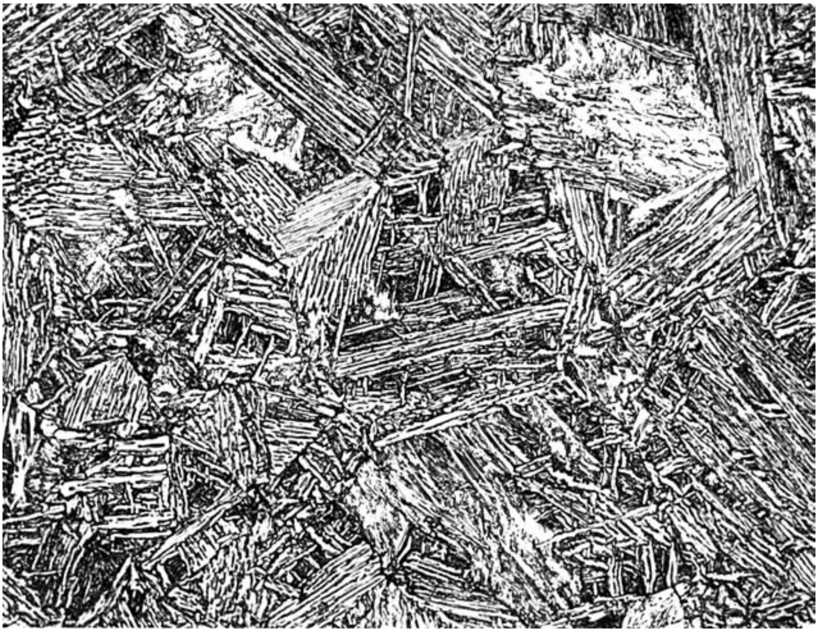


Fig. 8 Photomicrograph of a low-alloy steel showing a bainitic microstructure. 500X. 4% picral + 2% nital etch.

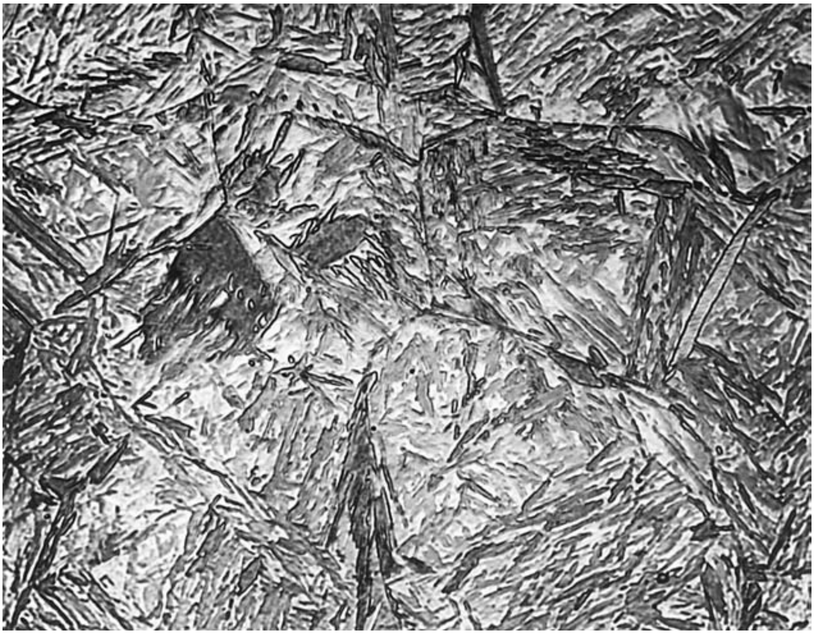


Fig. 9 Photomicrograph of a low-alloy steel showing a martensitic microstructure. 1000X. 4% picral + HCl and 10% sodium metabisulfate etch.

Continuous-Cooling Transformation Diagram. The other more useful form of a time–temperature transformation diagram is the continuous-cooling transformation (CT) diagram. This differs from the IT diagram in that it is constructed by cooling small specimens at various cooling rates and measuring the temperatures at which transformations start and finish using a device called a dilatometer (a machine that measures dilation). Each phase transformation undergoes a distinct volume change (positive on cooling and negative on heating) that can be measured by a sensitive length-measuring device in the dilatometer. A CT diagram has similar features to the IT diagram shown in Fig. 7 but is produced by continuous cooling rather than isothermal conditions. A continuous-cooling diagram is applicable for most industrial processes and should be used in lieu of an IT diagram. A CT diagram can also be constructed by quenching one end of a Jominy bar described below.

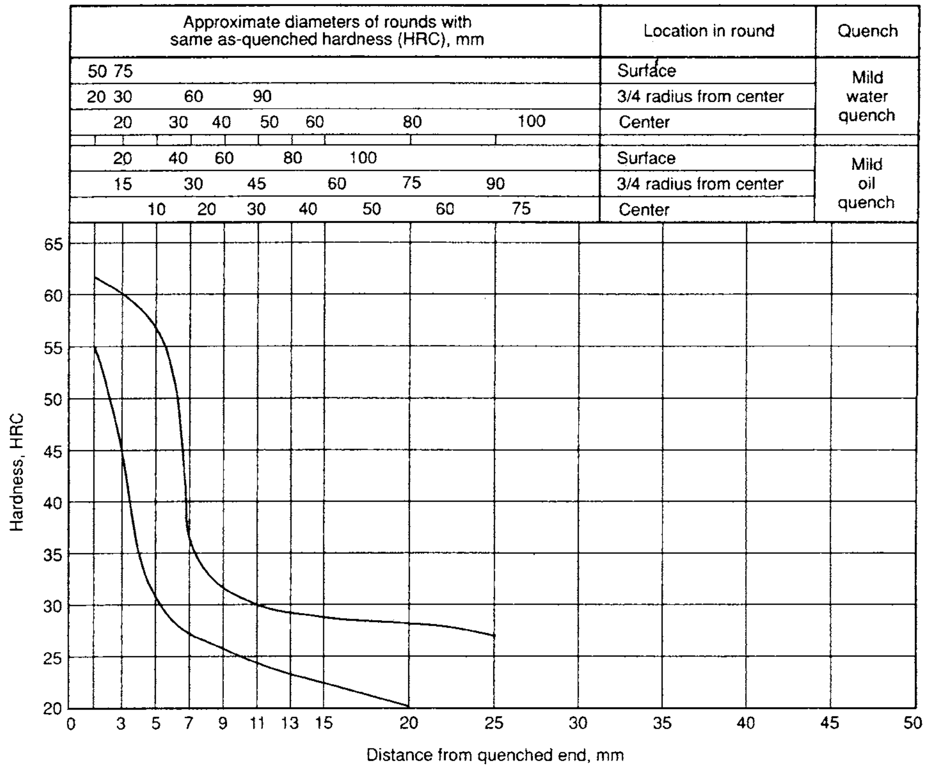
Hardenability Concept. In thick products, e.g., large-diameter bars, thick plate, and heavy forgings, the through-thickness properties are achieved through hardenability. Hardenability is the ability to induce depth of hardness in a steel product. The hardness level is obtained by controlling the amount of martensite in the microstructure. To increase the depth of hardness, certain alloying elements are added to the steel for increased hardenability. Elements, such as nickel, chromium, and molybdenum, shift the pearlite nose of the IT and CT diagrams to the right (longer times). With the nose out of the way on cooling, martensite can formed over a wider range of cooling rates when compared with a steel without alloying elements.

There is a fairly simple test to measure the hardenability of steel called the Jominy test. A 24.4-mm-diameter and 102-mm-long bar is austenitized to 845°C for 1 h and then water quenched at one end of the bar. The quenching takes place in a specially designed fixture where the bar is suspended in the vertical position and water is directed against the machined bottom end face of the bar. After quenching, parallel flats 0.38 mm deep are machined on opposite sides of the bar. Hardness is measured at 1.6-mm ($\frac{1}{16}$-in.) intervals from the quenched end. The hardness is plotted against depth from the quenched end to produce a hardenability curve or band. A hardenability band for medium-carbon SAE/AISI 1045 steel is shown in Fig. 10*a*. The two curves that form the band represent the maximum and minimum hardness values from many Jominy tests. To illustrate the concept of hardenability, compare the hardenability band for SAE/AISI 1045 steel to low-alloy SAE/AISI 4145 steel in Fig. 10*b*. These steels are similar except that the low-alloy steel has chromium and molybdenum additions as shown below:

C	*Mn*	*Si*	*Cr*	*Mo*
0.42/0.51	0.50/1.00	0.15/0.35	—	—
0.42/0.49	0.65/1.10	0.15/0.35	0.75/1.20	0.15/0.25

As can be seen from the hardenability bands, the higher manganese, chromium, and molybdenum additions in the SAE/AISI 4145 steel produced a much greater depth of hardness than the plain-carbon steel. For example, a hardness of HRC

Heat-treating temperatures recommended by SAE
Normalize (for forged or rolled specimens only): 870 °C (1600 °F)
Austenitize: 845 °C (1550 °F)

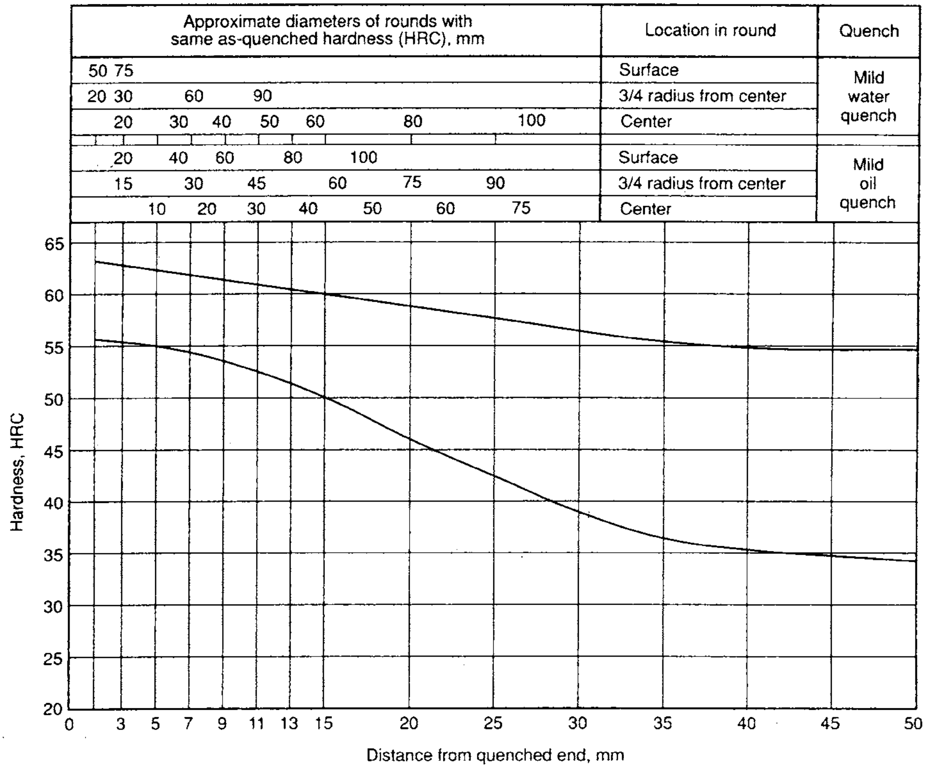


(a)

Fig. 10 Hardenability curves (*a*) SAE/AISI 1045 and (*b*) SAE/AISI 4145 showing depth of hardness with distance from the quenched end of a Jominy bar (*Source: ASM Handbook, Vol. 1, Properties and Selection: Irons, Steels, and High-Performance Alloys,* ASM International, Materials Park, OH 44073-0002, 1997, p. 487.)

45 (Rockwell *C* scale) was achieved at a depth of only 3–6.5 mm in the SAE/AISI 1045 steel compared with a hardness of HRC 45 at a depth of 21–50 mm in the SAE/AISI 4145 steel. This low-alloy steel has many times the depth of hardness or hardenability of the plain-carbon steel. This means that a hardness of HRC 45 can be achieved in the center of a 100-mm-diameter bar of SAE/AISI 4145 steel compared to a 10-mm-diameter bar of SAE/AISI 1045 steel (both water quenched). The depth of hardness is produced by forming martensite near the quenched end of the bar with mixtures of martensite and bainite further in from the end and eventually bainite at the maximum depth of hardness. Hardenability is important since hardness is roughly proportional to tensile strength. To convert hardness to an approximate tensile strength the conversion table in ASTM E140 can be used. A portion of this table is

Heat-treating temperatures recommended by SAE
Normalize (for forged or rolled specimens only): 870 °C (1600 °F)
Austenitize: 845 °C (1550 °F)



(b)

Fig. 10 (*Continued*)

Hardness			
Rockwell C Scale	Vickers	Brinell 3000-kg Load	Approx. Tensile Strength (MPa)
60	697	(654)	—
55	595	560	2075
50	513	481	1760
45	446	421	1480
40	392	371	1250
35	345	327	1080
30	302	286	950
25	266	253	840

This table also lists Vickers and Brinell hardness values, which are different types of hardness tests. It can be seen that a hardness of HRC 45 converts to an approximate tensile strength of 1480 MPa.

4 ROLE OF ALLOYING ELEMENTS IN STEEL

In the hardenability concept described in the previous section, alloying elements have a profound effect on depth of hardness. Alloying elements also change the characteristics of the iron–carbon diagram. For example, in the iron–carbon diagram (see Fig. 1) austenite cannot exist below the eutectoid temperature of 727°C. However, there are steels where austenite is the stable phase at room temperature, e.g., austenitic stainless steels and austenitic manganese steels. This can only be achieved through alloying. There are, however, special conditions where small amounts of austenite can be retained at room temperature during rapid quenching of low alloy steel. When this occurs, the austenite is too rich in alloying elements to transform at room temperature and is thus retained as small regions in a martensitic microstructure. Because of this, it is called retained austenite. The retained austenite can be transformed through tempering the steel.

In austenitic stainless steels, when nickel is added with chromium, the austenite phase field is expanded allowing austenite to be stable at room temperature. The popular SAE/AISI 304 austenitic stainless steel contains 18% Cr and 8% Ni. Austenitic manganese steel (Hadfield steel) contains 12% Mn with 1% C. The Mn and C allow austenite to be stable at room temperature. Because of this ability, nickel and manganese are, therefore, called austenite stabilizers. Other elements are ferrite stabilizers, e.g., chromium, silicon, and molybdenum. A ferrite-stabilizing element expands the ferrite phase field, and the austenite phase field is restricted within what is called a gamma loop (gamma, γ, is the symbol for austenite). A gamma loop can be seen in the iron–chromium equilibrium diagram in Fig. 11. The gamma loop is shown at the left side of the diagram. According to this diagram, iron–chromium alloys with 12.7% Cr or higher, the transformation from austenite (γ) to ferrite (α) does not occur and ferrite exists from room temperature to melting. Iron–chromium alloys make up an important class of stainless steels called ferritic and martensitic stainless steels.

Each particular alloying element has an influence on the structure and properties of steel. The following elements are important alloying elements in steel:

Carbon. Carbon is the most common alloying element in steel. It is inexpensive and has a strong influence on hardness and strength. It is the basic and essential alloying element in all plain-carbon, low-alloy, and tool steels. Carbon is an interstitial element that occupies sites between the larger iron atoms in the bcc and fcc lattices. The influence of carbon on the strength of iron can be seen in Fig. 12. Carbon can increase yield strength of pure iron (0% C) with a strength of about 28 to 190 MPa at 0.005% C, the maximum solubility of carbon at room temperature. This sevenfold increase in strength is due to interstitial solid solution strengthening. Any excess carbon, above 0.005% C, will form a iron carbide compound called cementite (Fe_3C). Cementite can exist as a particle, as a component of lamellar pearlite or as a proeutectoid network on prior austenite grain boundaries in hypereutectoid steel. Thus, carbon in the form of cementite has a further influence on the strength of steel, as seen in Fig. 13. In this plot, the steels between 0.1% C and 0.8% C contain about 10–100% pearlite. Yield strength peaks at about 425 MPa at 0.6% C whereas tensile strength (ultimate strength) increases to 790 MPa at 0.8% C. These properties are for carbon steels in the air-cooled condition. In a 0.8% C steel, a further

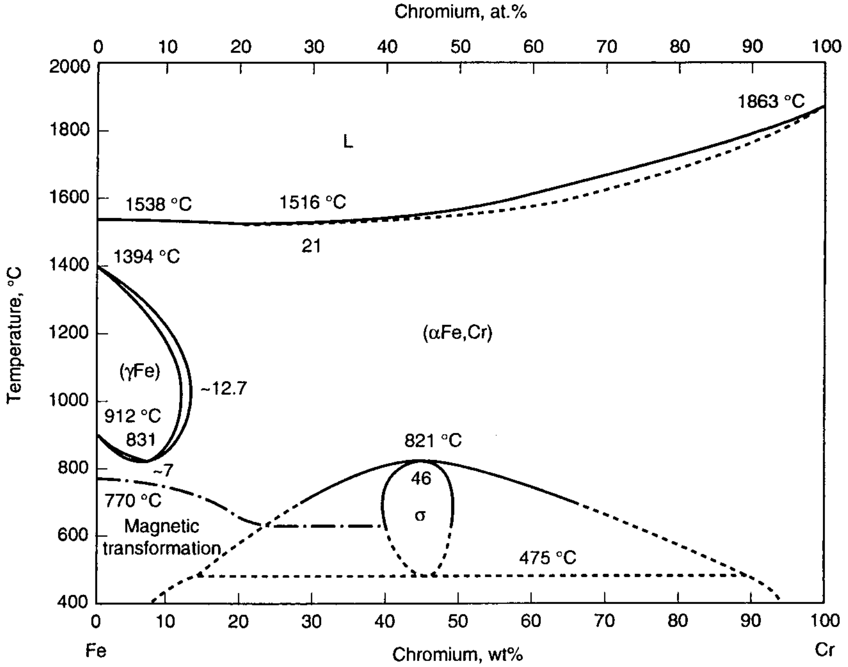


Fig. 11 Iron–chromium equilibrium phase diagram. (*Source: ASM Handbook, Vol. 20, Materials Selection and Design,* ASM International, Materials Park, OH 44073-0002, 1997, p. 365.)

increase in strength can be achieved if faster cooling rates are used to produce a finer pearlite interlamellar spacing. In a fully pearlitic, head-hardened rail steel (accelerated-cooled), the yield strength can increase to 860 MPa and tensile strength to 1070 MPa. Carbon also has a negative effect on properties, as seen in Fig. 13. For example, the percent reduction in area (as well as total elongation not shown) decreases with increasing carbon. The percent reduction in area is a

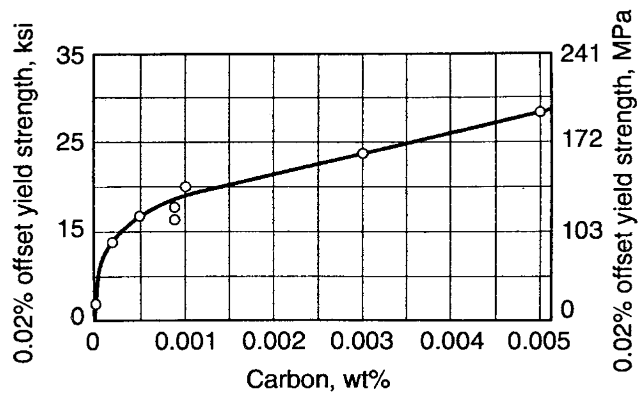


Fig. 12 Effect of carbon in solid solution on the yield strength of iron. (*Source: ASM Handbook, Vol. 20, Materials Selection and Design,* ASM International, Materials Park, OH 44073-0002, 1997, p. 367.)

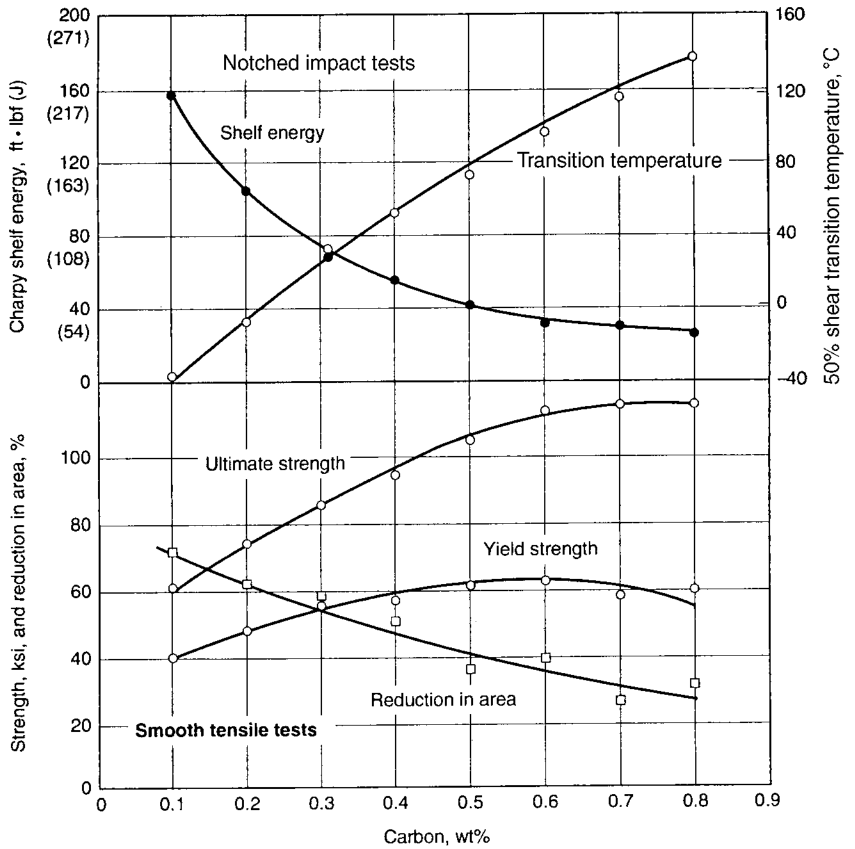


Fig. 13 Effect of carbon on the tensile and notched impact properties of ferrite–pearlite steels. (*Source: ASM Handbook, Vol. 20, Materials Selection and Design,* ASM International, Materials Park, OH 44073-0002, 1997, p. 367.)

measure of the cross-sectional area change in a tensile specimen before and after fracture. Notch toughness also decreases with carbon content as seen in the decrease in upper shelf energy and the increase in transition temperature. Shelf energy is the upper portion or upper shelf of a curve of absorbed energy plotted from a Charpy test.

Manganese. Manganese is also an essential element in all carbon, low-alloy, and alloy steels. Manganese has several roles as an alloying element. One role is to assure that all residual sulfur is combined to form manganese sulfide (MnS). Manganese is generally added to steel with a minimum manganese:sulfur ratio of 20:1. Without manganese the sulfur would combine with iron and form iron sulfide (FeS), which is a brittle compound that lowers toughness and ductility and causes a phenomenon called hot shortness. Hot shortness is a condition where a compound (such as FeS) or insoluble element (such as copper) in steel has a low melting point and thus forms an unacceptable cracklike surface condition during hot rolling. Another role of manganese is in strengthening steel.

Manganese is a substitutional element and can replace iron atoms in the bcc or fcc lattice. Each 0.1% Mn added to iron will increase the yield strength by about 3 MPa. Manganese also lowers the eutectoid transformation temperature and lowers the eutectoid carbon content. In large amounts (12% or higher), manganese is an austenite stabilizer in alloy steels and forms a special class of steels called austenitic manganese steels (also called Hadfield manganese steels). These steels are used in applications requiring excellent wear resistance, e.g., in rock crushers and in railway track connections where two rails meet or cross.

Silicon. Silicon is added to many carbon and low-alloy steels as a deoxidizer, i.e., it removes dissolved oxygen from molten steel during the steel-refining process. Oxygen is an undesirable element in steel because it forms oxide inclusions, which can degrade ductility, toughness, and fatigue resistance. Silicon has a moderate effect on strengthening steel but is usually not added for strengthening. Each 0.1% Si increases the yield strength of iron by about 8 MPa. It is a ferrite stabilizer and is found in some stainless steels. Silicon is also added to steel for enhanced electrical properties, e.g., iron–silicon transformer steels at 3.25% Si. These carbon-free steels have high magnetic permeability and low core loss.

Phosphorus. Phosphorus is considered a tramp or residual element in steel and is carefully restricted to levels generally below 0.02%. However, like carbon, phosphorus is an interstitial element that can substantially strengthen iron. For this reason, phosphorus is added to a special class of steels called rephosphorized steels for strength. Rephosphorized steels also have enhanced machinability.

Sulfur. Sulfur is also considered a tramp element in steel and is usually restricted to below about 0.02%. Although an element with a small atomic diameter, sulfur is not considered an interstitial alloying element because it is insoluble in iron. However, as in the case of phosphorus, sulfur is added to a special class of steels called resulfurized steels that have improved machinability. These steels are called free-machining steels.

Copper. In most steels copper is considered a tramp (residual) element and is restricted to levels below 0.04%. Copper, having a much lower melting point than iron, can create a detrimental steel surface condition known as hot shortness. Although not generally added to steel, there is a very special class of steels that contain high levels of copper to take advantage of the precipitation of copper particles during aging (a tempering process). These copper particles increase strength and hardness. Copper is also added to low-alloy steels for atmospheric corrosion protection (these steels are called weathering steels). One problem with copper in steel is that it cannot be oxidized and removed during steel refining. Thus, over time, the copper level of steel produced from steel scrap is slowly increasing.

Nickel. Nickel is an important element because of its positive effect on hardenability. Many important low-alloy steels contain nickel for this reason. Nickel, being a substitutional element in the iron lattice has a small effect on increasing yield strength. Nickel, being an austenite stabilizer, is also a vital element in austenitic stainless steels. Nickel is also important in steels for cryogenic applications, storage tanks for liquefied hydrocarbon gases. Nickel does not form a carbide and remains in solid solution.

Chromium. Like nickel, chromium has a positive effect on hardenability and is an important alloying element in many low-alloy steels. For corrosion resistance, chromium is present in all stainless steels as a solid solution element. In addition to hardenability and solid solution effects, chromium forms several important chromium carbides that are necessary for wear resistance in many tool steels and steels used for rolls in hot- and cold-rolling mills.

Molybdenum. Molybdenum is a potent hardenability element and is found in many low-alloy steels. Molybdenum, like chromium, forms several types of carbides that are important for wear-resistant applications, e.g., tool steels. Molybdenum is added to minimize temper embrittlement in low-alloy steels. Temper embrittlement occurs when low-alloy steels are tempered in the temperature range of 260–370°C. The embrittlement is caused by tramp elements such as phosphorus that accumulate at the prior austenite grain boundaries and thus weaken the boundaries. Adding molybdenum prevents the accumulation of these undesirable elements at the boundaries. Molybdenum also enhances the creep strength of low-alloy steels at elevated temperatures and is used in rotors and other parts of generators in electric power plants. Creep is an undesirable process that allows steel to slowly elongate or creep under load. Eventually the component will fail.

Vanadium. Although vanadium is a potent hardenability element, its most useful role is in the formation of a vanadium nitride and vanadium carbide (it can also be in a combined form of vanadium carbonitride). A very important role of vanadium is in microalloyed steels, also called high-strength, low-alloy (HSLA) steels. These steels are strengthened by precipitation of vanadium nitrides and vanadium carbides (vanadium carbonitrides). The formation of vanadium carbide is important for wear resistance. Vanadium carbide is much harder than iron carbide, chromium carbide, and molybdenum carbide. Vanadium is thus important in high-speed tool steels, which are used as drill bits that retain their hardness as the tool heats by friction.

Tungsten. Tungsten is not an addition to low-alloy steels but is a vital alloying element in high-speed tool steels where it forms hard tungsten carbide particles.

Aluminum. Aluminum is employed as a deoxidizer in steel and is generally used in conjunction with silicon (also a deoxidizer). A deoxidizer removes undesirable oxygen from molten steel. Once removed, the steel is called ''killed.'' Aluminum–silicon deoxidized (killed) steels are known as fine-grain steels. Another important role of aluminum is the formation a aluminum nitride (AlN) precipitate. Many steels depend upon the formation of AlN, especially steels used for sheet-forming applications requiring a high degree of formability such as parts that require deep drawing. These steels are called drawing-quality special-killed (DQSK) steels. The AlN precipitates help in the formation of an optimum crystallographic texture (preferred orientation) in low-carbon sheet steels for these deep-drawing applications. When aluminum combines with nitrogen to form AlN, the dissolved interstitial nitrogen is lowered. Lower interstitial nitrogen (interstitial nitrogen is also called free nitrogen) provides improved ductility. Aluminum can also substitute for silicon in electrical steels for laminations in electric motors and transformer cores.

Titanium. Titanium is a strong deoxidizer but is usually not used solely for that purpose. Titanium is important in microalloyed steels (HSLA steels) because of the formation of titanium nitride (TiN) precipitates. Titanium nitrides pin grain boundary movement in austenite and thus provides grain refinement. Another role of titanium is in steels containing boron where a titanium addition extracts nitrogen from liquid steel so that boron, a strong nitride former, remains in elemental form to enhance hardenability. Because of its affinity for both carbon and nitrogen, titanium is important in interstitial-free (IF) steels. Interstitial-free steels are an important class of steels with exceptional formability. Titanium, being a strong carbide former, is used as a carbide stabilizer in austenitic stainless steels (AISI type 321), ferritic stainless steels (AISI type 409, 439, and 444), and precipitation hardening stainless steels (AISI type 600 and 635).

Niobium (Columbium). Niobium is also important in microalloyed (HSLA) steels for its precipitation strengthening through the formation of niobium carbonitrides. Some microalloyed steels employ both vanadium and niobium. Because of its affinity for both carbon and nitrogen, niobium is an element found in some IF steels. Niobium is also added as a carbide stabilizer (prevents carbides from dissolving and reforming in undesirable locations) in some austenitic stainless steels (AISI type 347, 348, and 384), ferritic stainless steels (AISI type 436 and 444), and precipitation hardening stainless steels (AISI type 630).

Tantalum. Because of its affinity for carbon, tantalum, like niobium, is added as a carbide stabilizer to some austenitic stainless steels (AISI type 347 and 348).

Boron. On a weight percent basis, boron is the most powerful hardenability element in steel. A minute quantity of boron, e.g., 0.003%, is sufficient to provide ample hardenability in a low-alloy steel. However, boron is a strong nitride former and can only achieve its hardenability capability if in elemental form. Thus, boron must be protected from forming nitrides by adding a sufficient amount of titanium to first combine with the nitrogen in the steel.

Calcium. Calcium is a strong deoxidizer in steel but is not used for that purpose. In a aluminum-deoxidized (killed) steel, calcium combines with sulfur to form calcium sulfide particles. This form of sulfide remains as spherical particles as compared with manganese sulfide, which is soft and elongates into stringers upon hot rolling. Thus, steels properly treated with calcium do not have the characteristics associated with MnS stringers, i.e., property directionality or anisotropy.

Zirconium. Although expensive and rarely added to steel, zirconium acts like titanium in forming zirconium nitride participates.

Nitrogen. Nitrogen is added to some steels, e.g., steels containing vanadium, to provide sufficient nitrogen for nitride formation. This is important in microalloyed steels containing vanadium. Nitrogen, being an interstitial element like carbon, strengthens ferrite. A number of austenitic stainless steels contain nitrogen for strengthening (AISI type 201, 202, 205, 304N, 304LN, 316N, and 316LN).

Lead. Lead is added to steel for enhanced machinability. Being insoluble in iron, lead particles are distributed through the steel and provide both lubrication and chip breaking ability during machining. However, leaded steels are being discontinued around the world because of the environmental health problems associated with lead.

Selenium. Selenium is added to some austenitic stainless steels (AISI type 303Se), ferritic stainless steels (AISI type 430Se), and martensitic stainless steels (AISI type 416Se) for improved machined surfaces.

Rare Earth Elements. The rare earth elements cerium and lanthanum are added to steel for sulfide shape control, i.e., the sulfides become rounded instead of stringers. It is usually added in the form of mish metal (a metallic mixture of the rare earth elements).

Residual Elements. Tin, antimony, arsenic, and copper are considered residual elements in steel. They are also called tramp elements. These elements are not intentionally added to steel but remain in steel because they are difficult to remove during steelmaking and refining. Steels made by electric furnace melting employing scrap as a raw material contain higher levels of residual elements than steels made in an integrated steelmaking facility (blast furnace–BOF route). Some electric furnace melting shops use direct-reduced iron pellets to dilute the effect of these residuals.

Hydrogen. Hydrogen gas is also a residual element in steel and can be very deleterious. Hydrogen is soluble in liquid steel and somewhat soluble in austenite. However, it is very insoluble in ferrite and is rejected as atomic hydrogen (H^+). If trapped inside the steel, usually in products such as thick plate, heavy forgings, or railroad rail, hydrogen will accumulate on the surfaces of manganese sulfide inclusions. When this accumulation takes place, molecular hydrogen (H_2) can form and develop sufficient pressure to create internal cracks. As the cracks grow, they form what is termed hydrogen flakes and the product must be scrapped. However, if the product is slow cooled from the rolling temperature, the atomic hydrogen has sufficient time to diffuse from the product thus avoiding hydrogen damage. Also, most modern steelmakers use degassing to remove hydrogen from liquid steel.

5 HEAT TREATMENT OF STEEL

One of the very important characteristics of steel is the ability to alter the microstructure through heat treatment. As seen in the previous sections, many different microstructural constituents can be produced. Each constituent imparts a particular set of properties to the final product. For example, by quenching a steel in water, the steel becomes very hard but brittle through the formation of martensite. By tempering the quenched steel, some ductility can be restored with some sacrifice in hardness and strength. Also, superior wear properties can be obtained in fully pearlitic microstructures, particularly if an accelerated cooling process is employed to develop a fine interlamellar spacing. Complex parts can be designed by taking advantage of the formability and ductility of ferritic sheet steel through cold rolling and annealing. The amount of pearlite in ferritic steel can be adjusted by carbon content and cooling rate to produce a wide range of hardness and strength. In quenched and tempered steels, a bainitic microstructure has a unique combination of high strength and toughness. Thus steel, more than any other metallic material, can be manipulated through heat treatment to provide a multiplicity of microstructures and final properties. The common types of heat treatment are listed below:

Annealing (Full Annealing). One of the most common heat treatments for steel is annealing. It is used to soften steel and to improve ductility. In this process, the steel is heated into the lower regions of the austenite phase field and slow cooled to room temperature. The resulting microstructure consists of coarse ferrite or coarse ferrite plus pearlite, depending upon carbon and alloy content of the steel.

Normalizing. Steel is normalized by heating into the austenite phase field at temperatures somewhat higher than those used by annealing followed by air cooling. Many steels are normalized to establish a uniform ferrite plus pearlite microstructure and a uniform grain size.

Spheriodizing. To produce a steel in its softest possible condition, it is usually spheriodized by heating just above or just below the eutectoid temperature of 727°C and holding at that temperature for an extended time. This process breaks down lamellar pearlite into small spheroids of cementite in a continuous matrix of ferrite as seen in Fig. 14. To obtain a very uniform dispersion of cementite spheroids, the starting microstructure is usually martensite. This is because carbon is more uniformly distributed in martensite than in lamellar pearlite. The cementite lamella must first dissolve and then redistribute the carbon as spheroids whereas the cementite spheroids can form directly from martensite.

Process Annealing (Recrystallization Annealing). Process annealing takes place at temperatures just below the eutectoid temperature of 727°C. This treatment is applied to low-carbon, cold-rolled sheet steels to restore ductility. In aluminum-killed steels, the recrystallized ferrite will have an ideal crystallographic texture (preferred orientation) for deep drawing into complex shapes such as oil filter cans and compressor housings. Crystallographic texture is produced by developing a preferred orientation of the ferrite grains, i.e., the crystal axes of the ferrite grains are oriented in a preferred rather than random orientation.

Stress Relieving. Steel products with residual stresses can be heated to temperatures approaching the eutectoid transformation temperature of 727°C to relieve the stress.

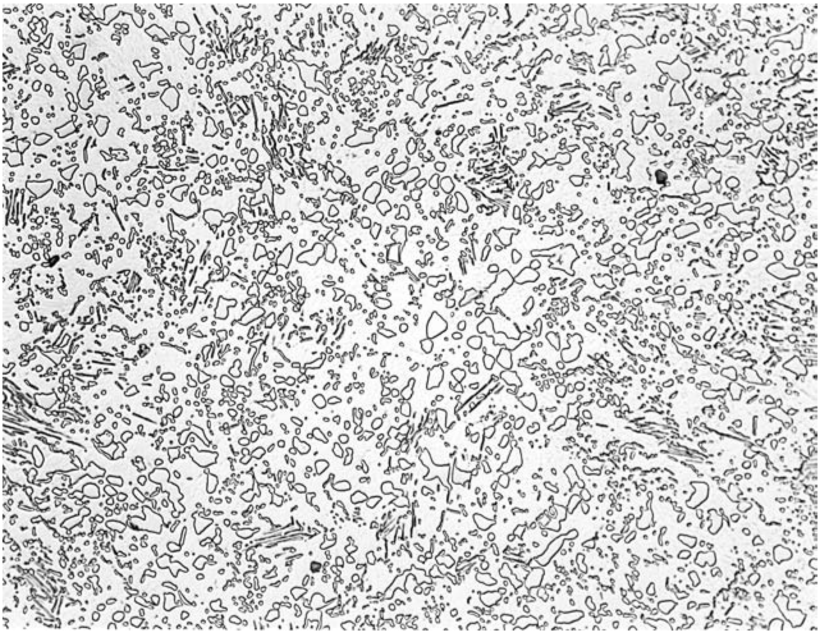


Fig. 14 Photomicrograph of a medium-carbon steel in the spheriodized condition. 500X. 4% picral + 2% nital etch.

Quenching. To produce the higher strength constituents of bainite and martensite, the steel must be heated into the austenite phase field and rapidly cooled by quenching in oil or water. High-strength, low-alloy (HSLA) steels are produced by this process followed by tempering. It must be noted that employing microalloying additions such as Nb, V, and Ti can also produce HSLA steels. These microalloyed steels obtain their strength by thermomechanical treatment rather than heat treatment.

Tempering. When quenched steels (martensitic steel) are tempered by heating to temperatures approaching the eutectoid temperature of 727°C, the dissolved carbon in the martensite forms cementite particles, and the steels become more ductile. Quenching and tempering are used in a variety of steel products to obtain desired combinations of strength and toughness.

6 CLASSIFICATION AND SPECIFICATIONS OF STEELS

Since there are literally thousands of different steels, it is difficult to classify them in a simple straightforward manner. However, some guidelines can be used. For example, steels are generally classified as carbon steel or alloy steel. A classification system was developed by the Society of Automotive Engineers (SAE) as early as 1911 to describe these carbon and alloy steels. The American Iron and Steel Institute (AISI) collaborated with SAE to refine the compositional ranges of the classification that are used today. Recently, a Unified Numbering System (UNS) was established that incorporates the SAE/AISI number.

Many steel products are purchased by specifications describing specific compositional, dimensional, and property requirements. Specification organizations such as ASTM (American Society for Testing and Materials) have developed numerous specifications for steel products and the testing of steel products. Some specific product user groups in the United States have developed their own specifications, e.g., the American Bureau of Ships (ABS) for ship plate and other marine products, Aerospace Materials Specifications (AMS) for aerospace applications, the American Railway Engineering and Maintenance of Way Association (AREMA) for rail and rail products, the Society of Automotive Engineers (SAE) for automotive applications, and the American Society of Mechanical Engineers (ASME) for steels produced to boiler code specifications. In Japan, there are standards developed by the Japanese Industrial Standards (JIS) Committee of the Ministry of International Trade and Industry. In the United Kingdom, there are the British Standards (BS) developed by the British Standards Institute. In Europe, Germany has the Deutsches Institut für Normung (DIN) standards, France the Association Francaise de Normalisation (AFNOR) standards, and Italy the Ente Nazionale Italiano di Unificazione (UNI) standards.

Specifications can be as simple as a hardness requirement, i.e., ASTM A1 for rail steel to elaborate compositional and property requirements as in ASTM A808 "High-strength low-alloy carbon–manganese–niobium–vanadium steel of structural quality with improved notch toughness." Describing specific specifications is beyond the scope of this handbook but many of the key sources can be found in the Bibliography at the end of this chapter.

6.1 Carbon Steels

Carbon steels (also called plain-carbon steels) constitute a family of iron–carbon–manganese alloys. In the SAE/AISI system, the carbon steels are classified as follows:

Nonresulfurized carbon steels	10xx series
Resulfurized steels	11xx series
Rephosphorized and resulfurized steels	12xx series
High manganese carbon steels	15xx series

A four-digit SAE/AISI number is used to classify the carbon steels with the first two digits being the series code and the last two digits being the nominal carbon content in points of carbon (1 point = 0.01% C). For example, SAE/AISI 1020 steel is a carbon steel containing 0.20% C (actually 0.18–0.22% C). The chemical composition limits for the above SAE/AISI 10xx series of carbon steels for semifinished products, forgings, hot- and cold-finished bars, wire, rods, and tubing are listed in *SAE Materials Standards Manual* (SAE HS-30, 1996). There are slight compositional variations for structural shapes, plates, strip, sheet, and welded tubing (see SAE specification J403). The *SAE Manual* gives the SAE/AISI number along with the UNS number. The carbon level spans the range from under 0.06% C to 1.03% C.

Because of the wide range in carbon content, the SAE/AISI 10xx carbon steels are the most commonly used steels in today's marketplace. All SAE/AISI 10xx series carbon steels contain manganese at levels between 0.25 and 1.00%. For a century, manganese has been an important alloying element in steel because it combines with the impurity sulfur to form manganese sulfide (MnS).

MnS is much less detrimental than iron sulfide (FeS), which would form without manganese present. Manganese sulfides are usually present in plain and low-alloy steels as somewhat innocuous inclusions. The manganese that does not combine with sulfur strengthens the steel. However, with the development of steelmaking practices to produce very low sulfur steel, manganese is becoming less important in this role.

The SAE/AISI 11xx series of resulfurized steels contain between 0.08 and 0.33% sulfur. Although in most steel, sulfur is considered an undesirable impurity and is restricted to less than 0.05%, in the SAE/AISI 11xx and 12xx series of steels, sulfur is added to form excess manganese sulfide inclusions. These are the free-machining steels that have improved machinability over lower sulfur steels due to enhanced chip breaking and lubrication created by the MnS inclusions.

The SAE/AISI 12xx series are also free-machining steels and contain both sulfur (0.16–0.35%) and phosphorus (0.04–0.12%). The SAE/AISI 15xx series contain higher manganese levels (up to 1.65%) than the SAE/AISI 10xx series of carbon steels.

Typical mechanical properties of selected SAE/AISI 10xx and 11xx series of carbon steels are listed in first part of the table on pp. 20–23, Section 4, of the *ASM Metals Handbook,* Desktop Edition, 1985, for four different processing conditions (as-rolled, normalized, annealed, and quenched and tempered). These properties are average properties obtained from many sources, and thus this table should only be used as a guideline. The as-rolled condition represents steel before any heat treatment was applied. Many applications utilize steel in the as-rolled condition. As can be seen from the aforementioned ASM table, yield and tensile strength is greater for steel in the normalized condition. This is because normalizing develops a finer ferrite grain size. Yield and tensile strength is lowest for steels in the annealed condition. This is due to a coarser grain size developed by the slow cooling rate from the annealing temperature. In general, as yield and tensile strength increase, the percent elongation decreases. For example, in the ASM table, annealed SAE/AISI 1080 steel has a tensile strength of 615 MPa and a total elongation of 24.7% compared with the same steel in the normalized condition with a tensile strength of 1010 MPa and a total elongation of 10%. This relationship holds for most steel.

Special Low-Carbon Steels

These are the steels that are not classified in the aforementioned SAE table or listed in the aforementioned ASM table. As mentioned earlier, carbon is not always beneficial in steels. These are special steels with carbon contents below the lower level of the SAE/AISI 10xx steels. There are a number of steels that are produced with very low carbon levels (less than 0.002% C), and all the remaining free carbon in the steel is tied up as carbides. These steels are known as IF steels, which means that the interstitial elements of carbon and nitrogen are no longer present in elemental form in the iron lattice but are combined with elements such as titanium or niobium as carbides and nitrides (carbonitrides). Interstitial-free steels are required for exceptional formability especially in applications requiring deep drawability. Drawability is a property that allows the steel to be uniformly stretched (or drawn) in thickness in a closed die without localized thinning and necking (cracking or breaking). An example of a deep-drawn part would be a compressor housing for a refrigerator. With proper heat treatment, IF steels develop a preferred crystallographic orientation that favors a high plastic anisotropy ratio or *r* value. High *r*-value steels have excellent deep drawing ability and these steels can form difficult parts. Another type of low-carbon steel is a special class called deep-quality special-killed (DQSK) steel. This type of aluminum-treated steel also has a preferred orientation and high *r* value. The preferred orientation is produced by hot rolling the steel on a hot strip mill followed by rapid cooling. The rapid cooling keeps the aluminum and interstitial elements from forming aluminum nitride particles (i.e., the Al and N atoms are in solid solution in the iron lattice). After rolling, the steel is annealed to allow aluminum nitride to precipitate. The aluminum nitride plays an important role in the development of the optimum crystallographic texture. DQSK steel is used in deep-drawing applications that are not as demanding as those requiring IF steel.

A new family of steels called bake-hardening steels also have a low, but controlled carbon content. These steels gain strength during the paint–bake cycle of automotive production. Controlled amounts of both carbon and nitrogen combine with carbonitride-forming elements such as titanium and niobium during the baking cycle (generally 175°C for 30 min). The precipitation of these carbonitrides during the paint–bake cycle strengthen the steel by a process called aging.

Enameling steel is produced with as little carbon as possible because during the enameling process, carbon in the form of carbides can react with the frit (the particles of glasslike material that melts to produce the enamel coating) to cause defects in the coating. Thus, steels to be used for enameling are generally decarburized in a special reducing atmosphere during batch annealing. In this process, the carbon dissipates from the steel. After decarburization, the sheet steel is essentially pure iron. Enamel coatings are used for many household appliances such as washers and dryers, stovetops, ovens, and refrigerators. Also, steel tanks in most hot-water heaters have a glass (or enameled) inside coating.

Electrical steels and motor lamination steels are also produced with as low a carbon content as possible. Dissolved carbon and carbides in these steels are avoided because the magnetic properties are degraded. The carbides, if present in the steel, inhibit the movement of the magnetic domains and lower the electrical efficiency. These steels are used in applications employing alternating current (AC) in transformers and electric motors. Most electric motors for appliances and other applications have sheet steel stacked in layers (called laminations) that are wound in copper wire. Electrical steels used for transformers contain silicon, which is added to enhance the development of a specific crystallographic orientation that favors electrical efficiency.

6.2 Alloy Steels

Alloy steels are alloys of iron with the addition of one or more of the following elements; carbon, manganese, silicon, nickel, chromium, molybdenum, and vanadium. The alloy steels cover a wide range of steels including low-alloy steels, stainless steels, heat-resistant steels, and tool steels. Some alloy steels, such as austenitic stainless steels, do not contain intentional additions of carbon. Silicon, when required, is added as a deoxidizer to the molten steel. Nickel provides strength and assists in hardening the steel by quenching and tempering heat treatment. This latter effect is called hardenability, which has been described earlier. Chromium is found in stainless steels for corrosion resistance. Chromium and molybdenum also assist in hardenability of the low-alloy steels. Vanadium strengthens the steel by forming precipitates of vanadium carbonitride. Vanadium is also a potent hardenability element.

Low-Alloy Steels

There is an SAE/AISI four-digit classification system for the low-alloy steels. As in the carbon steels, the first two digits are for the alloy class and the last two (or three digits) are for the carbon content. Because of the various combinations of elements, the system is more extensive than that used for the carbon steels. The general SAE/AISI classification system for low-alloy steels is as follows:

Manganese steels	13xx series
Nickel steels	23xx, 25xx series
Nickel–chromium steels	31xx, 32xx, 33xx, and 34xx series
Molybdenum steels	40xx, 44xx series
Chromium–molybdenum steels	41xx series
Nickel–chromium–molybdenum steels	43xx and 47xx series 81xx, 86xx, 87xx, and 88xx series 93xx, 94xx, 97xx, and 98xx series
Nickel–molybdenum steels	46xx and 48xx series
Chromium steels	50xx and 51xx series 50xxx, 51xxx, and 52xxx series
Chromium–vanadium steels	61xx series
Tungsten–chromium steels	71xxx, 72xx series
Silicon–manganese steels	92xx
Boron steels	xxBxx series
Leaded steels	xxLxx series

The boron-containing steels are low-alloy steels with boron added in the amount of 0.0005–0.003%. Boron is a strong hardenability element. The leaded steels contain 0.15–0.35% lead for improved machinability (however, lead is no longer favored as an alloying addition because of health concerns).

A table in the aforementioned SAE HS-30 lists the composition limits of most of the families of the SAE/AISI low-alloy steels listed above. These steels are supplied in the form of bar, plate, and forged products and are usually heat treated to obtain specific mechanical properties, especially high strength and toughness. Mechanical properties of selected SAE/AISI low-alloy steels in the as-rolled, annealed, normalized and quenched and tempered conditions are listed in the aforementioned ASM table. These properties are average properties and should only be used as a guideline. For example, SAE/AISI 4340 steel is usually heat treated by heating the component to 950–1000°C followed by quenching in oil. The component is then tempered at a temperature between 205–650°C. According to the aforementioned ASM table, this nickel–chromium–molybdenum steel in the quenched and tempered condition (tempered at 205°C) can achieve a yield strength of 1675 MPa and a tensile strength of 1875 MPa. Quenched and tempered low-alloy steels are used in a large number of applications requiring high strength and good toughness. Note that in the annealed condition, SAE/AISI 4340 steel has a yield strength of only 745 MPa.

Other Low-Alloy Steels

There are a number of important steels that do not fit into the SAE/AISI classification system described above. Such classes are the microalloyed steels also called high-strength, low-alloy (HSLA) steels, dual-phase steels, trip steels, and high-performance steels.

Microalloyed (High-Strength, Low-Alloy) Steels. Microalloying is a term applied to steels that contain small additions of alloying elements that retard austenite recrystallization and pin austenite grain boundary movement by the formation of small carbide and/or nitride precipitates. These elements include vanadium, niobium, and titanium. These HSLA steels are produced for a variety of plates, structural shapes, bars, and sheet applications with yield strength varying from 290 to 690 MPa. These steels are covered under numerous SAE and ASTM specifications. The SAE high-strength, low-alloy steels are covered under specifications J410, J1392, and J1442 and the ASTM high-strength, low-alloy steels are covered under various specifications including A242, A440, A441, A572, A588, A606, A607, A618, A633, A656, A690, A709, A714, A715, A808, A812, A841, A860, and A871. These HSLA steels have found wide application in areas such as bridge construction (structural beams), off-shore oil and natural gas platforms, ship hull and deck plate, and electrical transmission towers and poles. In the automobile, HSLA steels are used for safety (ultrahigh-strength impact door beams and energy-absorbing bumper assemblies) and for increasing fuel economy through thinner (lighter weight) chassis structural sections. Microalloyed HSLA steels are also employed in large-diameter gas transmission pipelines.

Dual-Phase Steels. A relatively recent development, dual-phase steels are produced by rapidly cooling a medium-carbon steel, containing vanadium or molybdenum, from the two-phase ferrite plus austenite region. The austenite transforms into islands of martensite (stabilized at room temperature by the V and Mo additions) in a ferrite matrix. Depending upon the alloy content, the martensite islands can also contain austenite that is retained below the transformation temperature (called retained austenite). Thus, dual-phase steel may contain both martensite and austenite as a second phase (called MA constituent). The unique characteristic of dual-phase steels is the continuous yielding behavior during deformation; i.e., there is a lack of a yield point during deformation. This provides increased uniform elongation and work hardening so that those components or parts produced from dual-phase steel actually gain strength during the forming operation. Dual-phase steels are being applied in applications such as automobile wheel rims and wheel disks. Because of their energy-absorbing characteristics, dual-phase steels are being used in critical locations of the automobile for safety to protect the occupants in the event of a crash.

Trip Steels. Similar to dual-phase steels, trip steels have emerged as an energy-absorbing high-strength steel for the automobile. The term "trip" is derived from the mechanism of *tr*ansformation *i*nduced *p*lasticity. These steels contain a high percentage of retained austenite (10–15%). The austenite transforms to martensite during the forming of the part, thus providing enhanced formability or transforms upon impact in a crash.

High-Performance Steels. There are a number of high-performance steels that are used in critical applications. These low-alloy steels, such as HY80 and HY100, are used in applications requiring high strength and excellent toughness. The "80" and "100" in the codes represents the minimum yield strength in ksi units. Another family of low-alloy steels is used in heat exchangers, high-temperature piping, and boiler applications. These steels like $2\frac{1}{4}$% Cr–1% Mo find wide use in these applications. Other high-performance steels are the Ni–Cr–Mo steels used as rotors for large steam generators and motors in electric power plants. These steels must withstand temperatures of superheated steam and must maintain high strength, superior toughness, as well as high fatigue strength and creep resistance. Ni–Cr–Mo–V steels are also used in pressure vessels in nuclear reactors.

Higher Alloy Steels

There is a distinction between the low-alloy steels described above and the higher alloy steels (usually containing over 8% alloying elements). The higher alloy steels include stainless steels, tool steels, heat-resistant steels, wear-resistant steels, and ultrahigh-strength steels.

Stainless Steels. Stainless steels are corrosion-resistant steels that contain at least 10.5% chromium. Chromium is unique in that it forms a passive layer on the steel surface that provides protection from corrosion. There are basically five types of stainless steels: austenitic, ferritic, duplex, martensitic, and precipitation hardening steels. These five types of stainless steel have a somewhat simplified classification system as follows:

Austenitic stainless steels with low nickel	2xx series
Austenitic stainless steels	3xx series
Ferritic stainless steels	4xx series
Duplex stainless steel	329
Martensitic stainless steels	4xx series
Precipitation strengthening stainless steels	6xx (xx-x PH)

The classification system is different for the stainless steels than the system for SAE/AISI low-alloy steels in that the last two digits (xx) do not represent the carbon content and have no particular compositional meaning. Unfortunately, the classification system has some confusion with the ferritic and martensitic stainless steels both of the 4xx series. The 2xx series of austenitic stainless steels were developed during the 1950s when nickel became scarce. In these steels, manganese and nitrogen were substituted for the lower nickel level in order to maintain strength. Each type of stainless steel is expanded upon below:

Austenitic Stainless Steels. Austenitic stainless steels have sufficient alloying to stabilize austenite at room temperature. These steels being austenitic are nonmagnetic. Austenitic stainless steels have excellent low-temperature toughness, weldability, and corrosion resistance. On the other hand, they have relatively low yield strength and can only be strengthened by cold working the steel, by precipitation hardening or by interstitial or substitutional solid solution strengthening.

The table on pp. 15.1–15.4 of the *ASM Metals Handbook,* Desktop Edition, 1985, lists the composition limits of the austenitic stainless steels. In general, the 3xx series are iron–chromium–nickel alloys that contain 16–26% chromium and 6–22% nickel. The popular type 304 austenitic stainless steel contains 18–20% Cr and 8–12% Ni and is often referred to as "18–8" stainless steel for the chromium and nickel content. There are many compositional variations of austenitic stainless steel steels. The following list summarizes these variations:

201	Low nickel replaced with manganese and nitrogen
202	Higher Mn than 201
205	Higher Mn and N than 202
301	Lower Ni and Cr to increase work-hardening ability
302	General-purpose 18–8 stainless steel
302B	Scaling resistance improved with Si
303	Enhanced machinability with a S addition
303Se	Improved machined surfaces with a selenium addition
304	Popular 18–8 stainless steel, lower C than 302
304L	Low-carbon 304 for improved corrosion resistance
304LN	Low-carbon 304 with nitrogen added for strength
304H	Higher carbon 304
304Cu	Copper added for improved cold working
304N	Nitrogen added for strength
305	Higher Ni for reduced work hardening
308	Higher Cr an Ni for weldability
309	High Cr and Ni for heat resistance
309S	Lower carbon 309
309Cb	Niobium (columbium) added
310	Higher Cr and Ni than 309 for improved heat resistance
310S	Lower carbon 310
310Cb	Niobium (columbium) added
314	Higher Si for improved heat resistance

316	Mo added for improved corrosion resistance
316F	Higher S and P for machinability
316L	Lower C for improved corrosion resistance and weldability
316LN	Lower C and higher nitrogen (for strength)
316H	Higher carbon 316
316N	Nitrogen added for strength
316Ti	Titanium added
316Cb	Niobium (columbium) added
317	Higher Cr and Mo for improved corrosion resistance
317L	Low-carbon 317 for improved weldability
321	Titanium added to minimize Cr carbide precipitation
330	High Ni to minimize carburization and improve thermal shock
347	Nb and Ta added to minimize Cr carbide precipitation
347H	Higher carbon 347
348	Ta and Co added for restricted nuclear applications
348H	Higher carbon 348
384	Higher Ni for decreased work hardening

The limiting of carbon is important in austenitic stainless steels. When heated, carbon forms chromium carbide that precipitates on the austenite grain boundaries and produces a condition known as sensitization. Because the chromium is tied-up as carbide, the chromium adjacent to the boundaries will be depleted in chromium and corrosion can take place. Sensitization is reversible by heating the steel to temperatures between 1040 and 1150°C followed by rapid cooling to room temperature. The high temperature dissolves the carbides and the rapid cooling prevents reprecipitation of the carbides. More on austenitic stainless steel can be found in the next chapter.

Ferritic Stainless Steels. The ferritic stainless steels are basically iron–chromium alloys with chromium ranging from 10.5 to 27%. The compositional limits for the ferritic stainless steels are listed in the aforementioned ASM table. Nickel is absent in ferritic stainless steels except for minor amounts, i.e., less than 1%, in some alloys. These steels have a microstructure of ferrite at room temperature and are magnetic. Type 409 stainless steel with the lowest chromium level (10.5–11.75%) is the least expensive of the ferritic stainless steel series and is used for automotive exhaust systems because it far outlasts carbon steel in that application. There are fewer variations of ferritic stainless steels than austenitic stainless steels. The ferritic stainless steels are listed below:

405	Low Cr with Al added
409	Low Cr, for automotive exhaust applications
429	
430	General-purpose ferritic stainless steel
430F	Free machining with higher S and P
430Se	Selenium added for improved machined surfaces

434	Mo added for improved corrosion resistance
436	Mo, Nb, and Ta added for corrosion and heat resistance
439	Low C, Ti added to minimize sensitization
442	Higher Cr for improved oxide scaling resistance
444	Low C, Mo for corrosion resistance, Ti and Nb for sensitization
446	Highest Cr for improved scaling resistance

Ferritic stainless steels are described in more detail in the next chapter.

Duplex Stainless Steels. Type 329 is an iron–chromium alloy with 2.5–5% nickel and 1–2% molybdenum that has a mixed (duplex) microstructure of approximately equal percentages of ferrite and austenite. There are many more duplex stainless steels that have priority compositions and trade names (see Bibliography at the end of chapter). The corrosion characteristics of these duplex stainless steels are similar to austenitic stainless steels. However, they have higher strength and better resistance to stress–corrosion cracking than austenitic stainless steels. Duplex stainless steels are discussed in the next chapter.

Martensitic Stainless Steels. To produce martensite in a stainless steel, the alloy must be transformed from the austenite phase field. According to the equilibrium phase diagram, this means that they have restricted chromium levels within the range required to form the gamma loop where austenite exists (see Fig. 11). The gamma loop is the region between 800 and 1400°C and 0 and 12.7% Cr in Fig. 11. Since austenite only exists in this restricted region, the steel must be heated within this temperature range and quenched to room temperature to form martensite. Martensitic stainless steels contain added carbon, which expands the gamma loop to allow higher chromium contents to be used. Because they can be heat treated, the martensitic stainless steels generally have higher strength than the austenitic and ferritic stainless steels. The martensitic stainless steels are listed below:

403	Select quality for highly stressed parts
410	General-purpose martensitic stainless steel
414	Ni added for improved corrosion
416	Higher P and S for improved machinability
416Se	Se added for improved machined surfaces
420	Higher C for increased strength
420F	Free machining with higher P and S
422	Mo, V, and W added for increased strength and toughness
431	Higher Cr, Ni added for improved corrosion resistance
440A	Highest Cr, C added for increased hardness
440B	Highest Cr, more C added for increased hardness/toughness
440C	Highest Cr, highest C for increased hardness/toughness
501	Low Cr, Mo added
502	Low C, Mo added

The compositional ranges for the martensitic stainless steels are shown in the aforementioned ASM table. Martensitic stainless steels are discussed in the next chapter.

Precipitation Hardening Stainless Steels. The precipitation hardening stainless steels are iron–chromium–nickel alloys that develop high strength and toughness through additions of Al, Ti, Nb, V, and/or N, which form precipitates during an aging heat treatment. The base microstructures of precipitation hardening stainless steels can be either martensitic or austenitic depending upon composition and processing. Some selected grades are listed below:

600	Austenitic grade with Mo, Al, Ti, V, and B added
630	Martensitic grade with Cu and Nb added
631	Austenitic grade with Al added
633	Austenitic grade with Mo and N added
635	Martensitic grade with Al and Ti added

The compositional ranges of the precipitation hardening stainless steels are listed in the aforementioned ASM table.

Other Stainless Steels. There are many stainless steels that do not fall within the AISI classification system. These steels have proprietary compositions and trade names. Details of many of these steels can be found in the next chapter and in the Bibliography at the end of this chapter.

Tool Steels. Tool steels are alloy steels that are used to cut or machine other materials. Tool steels contain various levels of Cr, Ni, Mo, W, V, and Co. The categories of tool steels are:

M series	Molybdenum high-speed steels
T series	Tungsten high-speed steels
Cr series	Chromium hot-work steels
H series	Molybdenum hot-work steels
A series	Air-hardening medium-alloy cold-work steels
D series	High-carbon high-chromium cold-work steels
O series	Oil-hardening cold-work steels
S series	Shock-resistant steels
L series	Low-alloy special-purpose tool steels
P series	Low-carbon mold steels
W series	Water-hardening tool steels

The compositional ranges of the various tool steels are listed in the table on pp. 758–759 of the *ASM Metals Handbook,* Vol. 20, 10th Edition, 1997. The high-speed steels are used in high-speed cutting tools such as drill bits. The hot-work tool steels are used in operations that utilize dies for punching, shearing, and forming materials at elevated temperatures, and the cold-work steels are used in similar operations at room temperature.

Heat-Resistant Steels. The upper temperature limit for use of carbon steels is about 370°C because of excessive oxidation and loss of strength. However, there are a number of alloy steels, called heat-resistant steels, that can be used at temperatures of 540–650°C. These steels include some of the ferritic stainless steels (405, 406, 409, 430, 434, and 439), quenched and tempered martensitic stainless steels (403, 410, 416, 422, and 431), precipitation hardening martensitic stainless steels (15-5 PH, 17-4 PH, and PH 13-8 Mo), precipitation hardening semiaustenitic stainless steels (AM-350, AM-355, 17-7 PH, and PH 15-7 Mo), and austenitic stainless steels (404, 309, 310, 316, 317, 321, 347, 202, and 216). In addition to the stainless steels, there are a number of proprietary alloys containing various levels of Cr, Ni, Mo, Nb, Ti, Cu, Al, Mn, V, N, or Si. The properties that are important to heat-resistant steels are creep and stress rupture. Creep is time-dependent strain occurring under stress. In more common terms creep is elongation or sagging of a part over time at elevated temperature. Stress rupture is a measure of the elevated temperature durability of material. These steels are generally specified under the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code.

Wear-Resistant Steels (Austenitic Manganese Steels). An important series of alloy steels are the austenitic manganese steels that contain 1.2% carbon and a minimum of 11% manganese. These steels, invented by Sir Robert Hadfield in 1882, are wear resistant and tough. Because they are difficult to hot work, these steels are usually cast into the final product form. The chemical compositional ranges for some selected austenitic manganese steels (ASTM A128) are listed below:

Grade	*C*	*Mn*	*Cr*	*Mo*	*Ni*	*Si*
A	1.55–1.35	11 (min)	—	—	—	1 (max)
B1	0.9–1.05	11.5–14	—	—	—	1 (max)
C	1.05–1.35	11.5–14	1.5–2.5	—	—	1 (max)
D	0.7–1.3	11.5–14	—	—	3–4	1 (max)
E1	0.7–1.3	11.5–14	—	0.9–1.2	—	1 (max)

The carbon addition is necessary to maintain an austenitic microstructure at room temperature. All grades must be heat treated by solution annealing at 1010–1090°C for 1–2 h per inch of thickness followed by rapid water quenching. Because these alloys work harden during use, they are used in applications involving earthmoving (bucket blades), mining and quarrying (rock crushers), and railway trackwork (frogs, switches and crossings).

Ultrahigh-Strength Steel

Maraging Steel. Another important series of alloy steels are the maraging steels. They are considered ultrahigh-strength steels because they can be heat treated to yield strength levels as high as 2.5 GPa. They also have excellent ductility and toughness. There are basically four grades that are produced to strength levels between 1.4 and 2.5 GPa.

Grade	*Ni*	*Co*	*Mo*	*Ti*	*Al*
18Ni (200)	18	8.5	3.3	0.2	0.1
18Ni (250)	18	8.5	5.0	0.4	0.1
18Ni (300)	18	9.0	5.0	0.7	0.1
18Ni (350)	18	12.5	4.2	1.6	0.1

The numbers in parentheses represent the nominal yield strength level in ksi. All maraging steels must be heat treated to achieve the desired properties. The heat treatment cycle for the 18Ni (200), 18Ni (250), and 18Ni (300) grades requires a solution treatment at 820°C for 1 h per 25 mm of thickness, cooling to room temperature and an aging treatment at 480°C for 4 h. The 18Ni (350) grade has an extended aging time of 12 h. The heat treatment develops a martensitic microstructure on cooling from austenite at 820°C. The aging step precipitates intermetallic compounds, e.g., Ni_3Mo that strengthen the martensitic matrix. Maraging steels can be machined before the aging treatment and have good weldability and excellent fracture toughness. They have found applications in missile and aircraft parts that require exceptional performance.

Music Wire. One of the strongest steel products commercially available is music wire. These wires can achieve levels of tensile strength approaching 5 GPa. The steel is basically SAE/AISI 1080. To obtain the ultrahigh-strength levels, rods of SAE/AISI 1080 are isothermally transformed to fine pearlite in a process known as patenting. The rods are then cold drawn to wire using large reductions in wire diameter through each die. The cold reduction forces the ferrite and cementite in the microstructure to align in a preferred orientation or fiber texture. The wires are used in musical instruments where they can be stretched under high tension to achieve certain musical notes. Ultrahigh-strength wires are also used to strengthen the rubber in automobile tires.

7 SUMMARY

Steel is one of the most versatile materials in today's society. It can be produced with a wide range of properties and is used in millions of applications. For example, stainless steels are used for their corrosion resistance, interstitial-free steels are used for their excellent formability characteristics, iron–silicon alloys are used for their electrical properties, austenitic manganese steels are used for their wear and abrasion resistance, microalloyed steels are used for their high strength, patented and cold-drawn eutectoid steel wires are used for their ultrahigh strength, dual-phase and trip steels are used for their energy absorption capability in a vehicle collision, and tool steels are used for their outstanding ability to cut and machine other materials. No other material can span such a range of properties and characteristics.

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CHAPTER 3

STAINLESS STEELS

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Stainless steels are those alloys of iron and chromium, with or without other elements, containing at least 11% chromium. This is the minimum amount of chromium necessary to form a stable, passive chromium oxide film. It is this film that is the basis for the corrosion resistance of all stainless, and most nickel base, corrosion-resistant alloys.

There are six basic classifications of stainless steels: ferritic, martensitic, martensitic age hardening, duplex austenitic–ferritic, and austenitic. The most commonly produced of these are the ferritics 409, for automotive applications, and 430 for corrosion-resistant/decorative uses, the martensitic grade 410, and the age-hardening martensitic 17-4PH®. Of the austenitic–ferritic duplex grades, alloy 2205 is the most broadly available. The two most used austenitic stainless grades are 304L, and 316L. A number of "superaustenitics" use nitrogen to maintain an austenitic structure with relatively high molybdenum, some 6%, and moderate nickel, 18–25%. Of the higher nickel grades 20Cb-3® stainless is used for sulfuric acid and general chemical processing. The most commonly used of the very high nickel alloys is C-276. The austenitic stainlesses form a continuum with the nickel base heat- and corrosion-resistant alloys. They are distinguished on the basis of nickel content by arbitrary or commercial definitions. There is no recognized metallurgical definition of where stainless ends and nickel-base begins.

Handbook of Materials Selection. Edited by Myer Kutz

1 EFFECT OF ALLOYING ELEMENTS

The corrosion behavior of the alloying elements in pure form influences the corrosion properties of the alloys.

Chromium is the first example, with outstanding corrosion resistance in the passive state. In solutions of neutral pH, dissolved oxygen from the air is sufficient to maintain passivity. But in low-pH solutions, stronger oxidizing agents must be present, and halogen or sulfuric acids absent, in order to stabilize the passive condition. Chromium metal is not resistant to corrosion by reducing acids.[1]

Some examples, from Uhlig,[1] of corrosion resistance of electrodeposited chromium:

Acid or Salt	Concentration (%)	Temperature °C	Temperature °F	Corrosion Rate (mm/yr)	Corrosion Rate (mils/yr)
Acetic	10	58	136	0.38	15
Ferric chloride	10	58	136	0.41[a]	16[a]
Formic	10	58	136	30	1200
Hydrobromic	10	58	136	4.7	186
Hydrofluoric	10	12	54	2.5	1000
Phosphoric	10	58	136	0.86	34
Sulfuric	10	12	54	0.28	11
Sulfuric	10	58	136	250	10,000[a]
Sulfuric	100	12	34	0.76	30
Sulfuric	100	58	136	1.8	69

[a] Pitting occurred, this number does not reflect uniform corrosion.

Three points can be made from this data. First, chromium as an alloying element is not particularly effective in promoting resistance to reducing or halogen acids. Second, in solutions of some halogen salts the passive layer was maintained by oxygen dissolved in the solution. Third, sulfuric acid behaves as a reducing acid in lower concentrations but as an oxidizing acid in concentrated form. When selecting alloys to resist sulfuric acid, one must bear this in mind.

Stainless steels containing only chromium and iron, specifically the ferritic and martensitic stainlesses, likewise have poor resistance to sulfuric acid solutions but may resist nitric acid. These chromium–iron alloys are not resistant to corrosion by halogen acids or by chloride salts. Those ferritic alloys that do have good to excellent chloride pitting resistance, such as E-BRITE® and AL29-4C®, gain that resistance by the addition of 1 and 4% Mo, respectively.

Austenitic nickel alloys with resistance to concentrated (oxidizing) sulfuric acid require high chromium, such as the Krupp VDM alloy 33, or silicon as in Haynes® Mickel alloy D-205, and the stainless grades A610, A611, and Sandvik® SX.

There are a few highly corrosion resistant nickel alloys with little or no chromium, including the various Ni–Mo “B” grades and the 67Ni–31Cu alloy 400. Excellent in reducing environments, they have almost no tolerance to oxidizing compounds in the environment. A newly developed nickel–molybdenum alloy, B-10, includes 8% Cr in its composition for limited resistance to low levels of oxidizers.

Molybdenum, in contrast to chromium, has very low resistance to oxidizing solutions but does resist reducing and halogen acids. Oxidizing acids such as nitric, aqua regia, and concentrated sulfuric acids readily dissolve molybdenum metal. Hydrofluoric acid does not affect Mo, and hot (110°C) hydrochloric acid attacks molybdenum metal only slowly.[1] In both stainless steels and nickel-base alloys, molybdenum as an alloying element is required for resistance to halogen acids and to pitting by acid or oxidizing chlorides. The amount used ranges from 2% in 316 L stainless up to 24–30% in alloys B through B-10. As an alloying addition, molybdenum improves the stability of the passive layer in the presence of halogens.

Tungsten at the 2–4% level is used, along with molybdenum, to improve chloride pitting corrosion resistance.

Nickel metal in general is attacked by oxidizing solutions, while reducing solutions are less aggressive. Some examples[1] follow:

Acid	Notes	Concentration (%)	Temperature °C	°F	Corrosion Rate (mm/yr)	(mils/yr)
Hydrochloric	Air saturated	10	30	86	2	80
Hydrochloric	N_2 saturated	10	30	86	0.25	10
Sulfuric	Aeration by convection	10	77	170	0.31	12.1
Sulfuric	Air saturated	10	82	180	4	160

Nickel metal is strongly attacked by phosphoric acid solutions containing ferric (oxidizing) salts, whereas it resists phosphoric acid solutions that are free of oxidizing compounds. Nickel metal resists neutral chloride solutions, such as sodium chloride, but is attacked by acid or oxidizing chloride salts. Alloys for use in reducing acids invariably have a considerable nickel content, ranging from as little as 8% in 304L to as much as 71% in alloy B-2.

Copper is generally resistant to reducing acid solutions containing only low levels of oxygen but is readily attacked by oxidizing acids. These include nitric, sulfurous, and concentrated sulfuric acids, as well as solutions containing oxidizing salts, such as ferric chloride. Copper in solution tends to reduce the corrosion rate of stainless alloys in (reducing) sulfuric acid. Those alloys intended for use in environments containing much sulfuric acid invariably have some copper as an alloy addition. These include 904L, 20Cb-3, 825, Nicrofer® 3127 hMo (alloy 31), and the Hastelloy® alloys G, G-3, and G-30.

Additions of some 4 or 5% silicon increase corrosion resistance to oxidizing environments. The silicon is primarily used in alloys meant to withstand concentrated, hence oxidizing, sulfuric acid. Such alloys in current production include A610, A611, Sandvik SX, and Haynes D-205. The use of silicon as an alloying element for corrosion resistance dates back to before Word War I. Although not a stainless, one of the oldest and most generally corrosion-resistant alloys ever developed is the 14.5% silicon cast iron, Duriron. A silicon oxide film is believed responsible for this grade's useful resistance to environments ranging from oxidizing to reducing. This includes seawater, organic and many inorganic acids, though not halogen acids. Lack of strength and ductility limit the cast iron's range of use.

When one speaks about the effect of this or that pure chemical on an alloy, one must emphasize that real industrial environments are complex mixtures of chemicals. These mixtures may behave in surprising ways; quite unlike what one might expect from the behavior of alloys in pure, laboratory-controlled environments. Corrosion rates depend not only upon the concentrations of various chemicals but also on the temperature. The temperature of liquid inside a vessel is one point that can be measured, but the temperature at the surface of submerged heating coils in that vessel is another, and higher value. Likewise the concentration of, say an acid, in the vessel is not the same as the concentration at the point where that acid is introduced to the mixture.

The most commonly used corrosion-resistant alloys are the stainless steels 304 (18% Cr 8% Ni, commonly known as 18–8 stainless) and 316 (17% chromium 11% nickel 2% molybdenum). The more corrosion-resistant nickel alloys, such as C-276, have much higher levels of nickel, 57%, and molybdenum, 15.5%. Commercially pure nickel, and nickel–copper alloys are used for special environments.

Oxidizing and reducing environments are defined chemically with respect to whether hydrogen is oxidized or reduced under the environment in question. In an oxidizing environment, hydrogen will only be present chemically combined with some other element, for example, with oxygen to form H_2O. In a reducing environment, that H^+ will be reduced to hydrogen gas, H_2.

Common oxidizing chemicals are nitric acid, HNO_3, and certain salts such as ferric chloride, $FeCl_3$, and cupric chloride, $CuCl_2$. The ferric and cupric ions are at a relatively high valences, +3 and +2, respectively, and readily accept electrons, or oxidize, other materials, to get their own valences reduced to a more stable level. Sulfuric acid, H_2SO_4, is normally a reducing acid. At high concentrations, above about 95%, sulfuric acid changes its character and becomes an oxidizing acid. Of course, dissolved oxygen contributes to the oxidizing character of an environment. To some extent so does dissolved elemental sulfur.

To resist oxidizing conditions an alloy must contain some amount of chromium. For oxidizing acid service simple materials such as 304 (18% Cr 8% Ni) or 310 (25% Cr 20% Ni) are often used. An unusually high level of chromium, 33%, is present in a newly developed alloy, UNS R20033, meant to resist very oxidizing acids. In any of these alloys the nickel content is necessary to make a stable austenitic alloy, but it does not contribute specifically to oxidizing acid resistance. Small additions of molybdenum or copper may be tolerated in these alloys to enhance resistance to chlorides or sulfuric acid. But neither Mo nor Cu themselves are helpful in resisting strongly oxidizing chemicals.

A common, and severe, test for resistance to oxidizing acids is boiling 65% nitric acid. The test is run for five periods of 48 h each, specimens being weighed after each test period, and the results averaged. This test is a good measure of resistance to intergranular corrosion in a sensitized alloy, as well as to general corrosion in nitric acid. Test results[2] show 2205 0.13–0.20 mm/yr, which is good, 304 0.23 mm/yr, and RA333®, which has been stabilize annealed 1700°F, at 0.29 mm/yr. In the case of RA333 it is the high chromium that helps, in spite of 3% Mo. Other molybdenum bearing grades do not fare so well, 316L (2% Mo) at 0.87 mm/yr after only 24 h, AL-6XN® (6.3% Mo) at 0.74 mm/yr, 625 (9% Mo) at 0.76 mm/yr, and C-276 (15.5% Mo) at 0.74 mm/yr. These results do not mean that one cannot successfully use a higher Mo alloy in the presence of any nitric acid at all. They do indicate that high-molybdenum alloys may not behave at all well in hot, concentrated oxidizing industrial environments.

One cannot readily find boiling 65% nitric acid (ASTM A262C) data for the 66% Ni–31% Ni alloy 400 (Monel® 400) or for the assorted B alloys—B, B-2, B-3, or B-4. Their corrosion rates in nitric acid are simply too high for the test to have any practical value. Alloys 400, B, and B-2 have no deliberate chromium addition, B-3 and B-4 only about 1.3% Cr. These grades have excellent resistance to various reducing environments, but because there is essentially no chromium present, they will literally dissolve in nitric acid. Likewise, they are attacked by ferric, cupric, and chlorate ions, and even dissolved oxygen in HCl.

The common "reducing" acids are sulfuric under about 95%, phosphoric (H_3PO_4), and hydrochloric. Of these by far the most corrosive is HCl, phosphoric being the less troublesome. Because reducing industrial environments often do contain some oxidizing salts or oxygen from the air, most alloys used to withstand reducing chemical environments will contain chromium, at least 15%. The alloy additions used to resist the reducing components of the environment are nickel (Ni), molybdenum (Mo), and copper (Cu).

In sulfuric acid some amount of copper is usually used, such as in 20Cb-3 stainless, 904L, or 825. Even copper salts in the acid will reduce corrosion attack of stainless. 20Cb-3 uses carefully balanced proportions of Cu and Mo to resist sulfuric acid corrosion.

2 SOME FORMS OF CORROSION

2.1 General Corrosion

This is the most common form of corrosion, accounting for the greatest tonnage loss of metal. It is characterized by relatively uniform attack of the entire area exposed to the corrosive environment. The passive film slowly dissolves but continually reforms. Since the attack is linear with time, the life of equipment subject to general corrosion is reasonably predictable. If the passive film is locally disrupted, as by chlorides, corrosion modes such as pitting, crevice, and stress corrosion may occur. These are more difficult to predict and tend to cause premature equipment failures. Erosion may also remove the passive film and contribute to much higher than expected general corrosion rates.

Stainless steel passivates simply by being exposed to air. A metallographic specimen of AL-6XN, for example, must be etched immediately after polishing. Otherwise it will passivate in air so that a uniform etch cannot be achieved. Passivation in acid is not required. But, during normal fabrication practice, enough iron is picked up to cause surface rusting in damp weather. A treatment in nitric–hydrofluoric acid may be used to remove this surface iron contamination.

Uniform corrosion rates may be stated as an average metal thickness loss with time, mils per year, or millimeter per year. A convenient rating for metals subject to uniform attack based on corrosion rates follows:

Excellent—rate less than 5 mils/yr (0.13 mm/yr). Metals suitable for making critical parts.

Satisfactory—rate 5 to 50 mils/yr (0.13–1.3 mm/yr). Metals generally suitable for noncritical parts where a higher rate of attack can be tolerated

Unsatisfactory—rates over 50 mils/yr (1.3 mm/yr). Metals usually not acceptable in the environment.

An approximate ranking of a few common alloys by increasing resistance to general corrosion would be 304L, 316L, 20Cb-3/825, AL-6XN, 625, and C-276. Alloy selection does depend upon the exact corrosive environment in question. Some specific examples include hot concentrated caustic, where commercially pure nickel or the 76% nickel alloy 600 are used. For sulfuric acid alloys 20Cb-3 or 825 are usually chosen—however, if chlorides are present in the acid, one of the 6% molybdenum grades such as AL-6XN would be preferred. AL-6XN is used for organic acids, such as napthenic acid in refinery service. For nitric acid service chromium is beneficial, molybdenum not. Alloys commonly selected include 304L or a low carbon version of 310. RA333 is used when the same piece of equipment must see very high temperatures, in the red heat range, in one zone and aqueous corrosion in another.

2.2 Stress–Corrosion Cracking

For just about every alloy there is some chemical environment that, combined with stress, will cause cracking. For brass that environment is ammonia or other nitrogen compounds. The source of stress is usually residual forming and welding stresses, which may reach the yield point of the material. Operating stress is rarely the issue.

For austenitic stainless steels chlorides are the major cause of stress–corrosion cracking (SCC). An example is hot potable water under heat-transfer conditions, which permit chlorides to concentrate locally. Susceptible alloys include 304L, 316L, 321, and 347. Some 95% of 316L chemical plant equipment failures may be attributed to chloride stress–corrosion cracking. The chlorides concentrate from trace amounts present in steam for heating, or the cooling water in heat exchangers, as well as from the product.

Chloride SCC occurs most quickly in stainless steels with about 8–10% nickel, alloys with much lower, or much higher, nickel content being less susceptible. As thermal stress relief is rarely practical with stainless fabrications, the metallurgical solution is a change in alloy. Nickel-free ferritic steels, such as E-BRITE are highly resistant to chloride SCC but impractical to fabricate into a vessel.

The traditional solution in the United States has been to go to a higher nickel alloy. Alloys with about 30% or more nickel are generally considered to be good engineering solutions to most chloride SCC problems, although they will crack under very severe conditions. 20Cb-3 at about 34% and 825 at 40% nickel have long been chosen for this service. Likewise the fine-grained Incoloy® 800, UNS

N08800, 31%Ni, had been used in years past. However, this low-carbon, fine-grained version of 800 is now rarely available. Regardless of what it is called, "800" today usually is 800HT, UNS N08811, a higher carbon, coarse-grained version. This grade is designed to maximize creep rupture strength for high-temperature applications.

Since the mid-1980s the 6% molybdenum superaustenitics have become available. Grades such as 254 SMO® with only 18% nickel, or AL-6XN at 24% nickel, have been used effectively to resist chloride SCC. The material cost of the "6-moly" grades is approximately three times that of 316L stainless. Although lower in nickel, molybdenum contents above 2% tend to decrease susceptibility of austenitic stainless to chloride SCC.[3]

For greater resistance to both corrosion and chloride SCC the most used grade has been C-276, at 57% Ni 15.5% Mo. There are now a number of alloys in this class, including ALLCORR™, C-22, Inconel® 686, C-2000, 59, and a new Japanese grade, MAT 21. These very high nickel alloys can easily reach five times or more the cost of 316L stainless. They are metallurgically excellent solutions to chloride SCC, particularly in severe environments. However, they are expensive choices for service conditions under which 316L lasted a few years before cracking. There is a less expensive choice, one which has long been used in Europe. That is a duplex stainless steel, which is about half austenite and half ferrite. Duplex stainless steel is a practical solution to most 304L or 316L SCC failures. The most commonly available duplex in North America is 2205, at a cost roughly 20% above that of 316L.

Other forms of stress–corrosion cracking in stainless steels include caustic cracking and polythionic acid stress–corrosion cracking. Caustic may crack carbon steel as well as stainless. High nickel alloys, such as alloy 600 or, better, commercially pure nickel (UNS N02201) are used.

Polythionic acid stress–corrosion cracking (PASSC) is caused by sulfur compounds in the environment and most often encountered in refineries. Any stainless or nickel alloy that has been sensitized can be subject to PASCC. High nickel does not help, even 600 alloy will crack when sensitized.[4] To resist this form of SCC the alloy must contain a strong carbide-forming element, or "stabilizing" element, such as columbium or titanium. Examples include 321, 347, 20Cb-3, 825, and 625. In addition the alloy must be given a stabilizing anneal so that the carbon is effectively combined with the Cb or Ti. RA333, because of its tungsten and molybdenum content, resists PASCC when stabilize annealed about an hour at 1700°F (927°C).

Normally, 304H or 316H would be quite sensitive to polythionic acid stress cracking, as these higher carbon, solution-annealed alloys readily sensitize. The matter has been addressed at one refinery by fabricating the equipment from one of these H-grade stainless steels, then stabilize annealing the completed fabrication. A temperature of about 1650°F (900°C) for a minimum of 1 h is used. This does precipitate carbides at the grain boundaries, but temperature is high enough to permit chromium to diffuse back into the Cr-depleted grain boundary zone. In addition, this treatment relieves over half of the residual fabricating stress, thus reducing susceptibility to chloride stress–corrosion cracking as well.

2.3 Pitting Corrosion

Pitting is an extremely localized form of corrosion that results in holes in the metal. Although total metal loss may be small, the equipment may be rendered useless because of perforation. Pitting usually requires a long initiation period before attack is visible. Once a pit has begun, the attack continues at an accelerating rate. Pits tend to grow in a manner that undermines or undercuts the surface. Typically a very small hole is seen on the surface. Poking at this hole with a sharp instrument may reveal a rather cavernous hole under what had looked like solid metal. In effect, a pit may be considered a self-formed crevice. Pitting attack increases with temperature.

Chloride solutions are the most common cause of pitting attack on stainless steels and nickel alloys. The alloying additions of molybdenum, nitrogen, and, to some extent, chromium, all contribute to pitting resistance. A laboratory measure of resistance to pitting corrosion is the critical pitting temperature, or CPT, which is the highest temperature at which an alloy resists pitting in a given environment. Alloy ranking with respect to chloride pitting resistance would be 304L (0% Mo, poor), followed by 316L (2% Mo), then four austenitics each with about 3% molybdenum, 20Cb-3/825/317L/RA333, the duplex 2205 (3% Mo 0.16% N), AL-6XN (6.3% Mo 0.22% N), 625 (9% Mo), and C-276 (15.5% Mo). Alloys AL-6XN and higher have chloride sufficient resistance to be used in hot seawater service. The lower molybdenum grades, including 2205 with 3% Mo, are unsuitable for use in seawater.

2.4 Crevice Corrosion

Crevice corrosion, more so than pitting, is the limiting condition that often prevents the use of conventional austenitic stainless in chloride environments. The attack usually occurs in small volumes of stagnant solution under gasket surfaces, lap joints, marine fouling, solid deposits, and in the crevices under bolt heads and the mating surfaces of male and female threads. The mechanism involves oxygen depletion in the crevice, followed by chloride ion concentration and increase in acidity (decrease in pH) within the crevice. In a neutral, pH 7, chloride solution service, the liquid within a crevice may contain 3–10 times as much chloride as the bulk solution, and have a pH of only 2–3. Susceptibility to crevice corrosion increases rapidly with temperature. Molybdenum and nitrogen additions to nickel–chromium–iron alloys improve their resistance to crevice corrosion. Together with the use of appropriate materials, design practice to minimize crevices and maintenance procedures to keep surfaces clean are required to combat the problem.

The usual laboratory measure of resistance to crevice corrosion is the critical crevice corrosion temperature, or CCCT, which is the highest temperature at which an alloy resists crevice corrosion in a given environment. For a given environment the CCCT is usually significantly lower than the CPT. Crevice corrosion resistance as measured by the ferric chloride test relates, to a degree, to performance in seawater. Here are the results for a number of alloys[2]—temperature for initiation of crevice corrosion in ferric chloride ($FeCl_3{\cdot}6H_3O$), 10% $FeCl_3{\cdot}6H_2O$, per ASTM G48 Practice B, (PRE) $N = \text{Cr} + 3.3\%\ \text{Mo} + 30\%\ N$:

Alloy	Mo (%)	Temperature °C	°F	Pitting Resistance Equivalent (PRE), *N*	Ref.
316L	2.1	−3	27	23	5
825	2.7	−3	27	30	5
317L	3.2	2	35	29	5
2205	3.1	20	68	38	5
317LMN	4.4	20	68	34	5
28	3.5	24	75	38	6
904L	4.4	24	75	35	5
904L	4.4	25	77	35	7
G	6.5	30	86	43	5
28	3.5	35	95	39	7
2507	4.0	35	95	47	7
1925hMo®	6.2	40	104	47	7
33	1.4	40	104	50	7
AL-6XN	6.2	43	110	48	5
625	9.0	45	113	51	5
625	9.0	55	131	51	6
31	6.5	55	131	54	6
G-30®	5.5	50	122	48	7
C-276	15.4	55	130	66	5

2.5 Intergranular Corrosion

Intergranular corrosion consists of localized attack along the grain boundaries of the metal. Sensitization to this attack in stainless or nickel alloys is caused by precipitation of chromium-rich carbides in the grain boundaries, at a temperature low enough that a chromium-depleted zone forms. This precipitation most commonly occurs from the heat of welding. It may also result from a slow cool after annealing, or from prolonged exposure to intermediate temperatures, roughly 850–1470°F (450–800°C), in service. For exposures at very long times, or around the high end of this range, diffusion of chromium back into the depleted zone will restore the corrosion resistance.

A most effective means of combating intergranular corrosion is to restrict the carbon content of the alloy. In the stainless "L" grades 0.03% maximum is considered sufficient. High chromium and molybdenum additions, as in AL-6XN, also reduce the chance of intergranular attack. Another approach is to add columbium or titanium to tie up the carbon, the same as is done to resist polythionic acid stress–corrosion cracking. 20Cb-3 stainless takes both approaches, being melted to low carbon, as well as having a columbium addition.

2.6 Galvanic Corrosion

An electrical potential, or voltage, difference will exist between two different metals that are in electrical contact and immersed in a corrosive solution. This potential difference causes current to flow and the less noble, or more anodic, metal suffers increased corrosion rate. The severity of attack depends upon the relative voltage difference between the metals, the relative exposed areas of each, and the particular corrosive environment.

The most common example is the old-fashioned flashlight battery, or dry cell. It has a shell of zinc metal (less noble, or anodic), filled with a moist, corrosive chloride paste that conducts electricity. The center post is made of graphite, which is quite noble (cathodic, does not tend to corrode). The potential (voltage) difference between zinc and graphite happens to be about 1.5 V. When an electric connection is made in a flashlight, the zinc corrodes, giving up electrons, which flow through the lightbulb toward the graphite cathode, the positive pole. In this case, because generating electricity is the point, no one minds that the zinc corrodes and gets used up.

The ratio of cathodic (noble) to anodic areas is an important factor in galvanic corrosion. An undesirable situation is a large cathode connected to a small anode, or less noble metal. This can develop high current density, hence severe corrosion, at the anode. In that common zinc dry cell the zinc anode has a much larger area than the graphite cathode, so it has some useful working life before corroding through the zinc case.

For example, a large area of stainless in contact with a small surface area of carbon steel is undesirable. The potential difference will tend to corrode the carbon steel, and the very large area of stainless will make that corrosion occur quickly. The reverse condition is preferred. That is, a small area of stainless (or more noble metal) may be coupled with a much larger area of carbon steel (anodic) without significant problems. "Significant" depends upon the application. In the past, when ferritic stainless trim was used on carbon steel automobile bodies, the steel would tend to corrode most severely underneath the trim. In part that was because the crevice trapped salt, but it was accentuated by the galvanic difference between ferritic stainless and carbon steel.

There is some potential difference among the various stainless and nickel alloys. In practice, galvanic corrosion is rarely a problem among these various alloys. There is, however, a significant potential difference between copper alloys and stainless. So long as the stainless is passive (not actively corroding) it is enough more noble than copper to corrode the copper alloy. An example is when a heat exchanger with a Muntz metal (60% Cu, 40% Zn) tubesheet is retubed with AL-6XN alloy instead of the original copper alloy tubes. The potential difference is enough to corrode the copper alloy tubesheet. One ought either to replace the tubesheet as well, with stainless, or retube using a copper base alloy.

Graphite is at the noble end of the galvanic series. If graphite is in contact with stainless or nickel alloys in a corrosive environment, those alloys may corrode preferentially.

Galvanic effects have a positive side and may be used to protect equipment from corrosion, a common example being a zinc coating on steel. The zinc corrodes preferentially, and in doing so protects the steel from corrosion (rusting). Zinc or magnesium anodes are often connected to equipment from chemical process to steel ship hulls to protect them from corrosion.

3 AOD, DUAL CERTIFICATION, AND CHEMISTRY CONTROL

Most stainless steels, and a few higher nickel alloys, are available with different levels of carbon. For resistance to intergranular corrosion, a low carbon is preferred, usually 0.03% carbon maximum in stainless. Such a stainless is referred to as an "L" grade, e.g., 304L and 316L. With respect to aqueous corrosion resistance, the lower the carbon, the better. For high-temperature service the opposite is true, and some minimum amount of carbon is required for both tensile and creep rupture strength.

The argon–oxygen decarburization (AOD) process for refining stainless steel was introduced in the 1970s. This made profound changes in how existing grades were produced, as well as permitting totally new grades to be developed. Three of these changes are worth discussing—carbon, sulfur, and precise control of chemistry.

Prior to the AOD, carbon could not be removed in the refining process without also removing chromium. Low-carbon grades could only be produced by starting with low-carbon raw materials, specifically low-carbon ferrochrome. The expense of low-carbon ferrochrome meant that the L grades were inherently more expensive. The AOD now permits refining carbon to very low levels, even with starting stock of higher carbon.

Industrywide specifications such as the American Society for Testing and Materials (ASTM) were written prior to the introduction of this new melting process. For example, ASTM A 240 for 304 stainless, UNS S30400, calls out 0.08% carbon maximum, no minimum, 30,000 psi minimum yield strength. Low carbon 18–8, 304L, S30403 is limited to 0.03% carbon, with a consequent lower limit for yield strength, 25,000 psi minimum. In addition, there is a 304H, meant for high-temperature use, with carbon specified as a range, 0.04–0.10%, and annealing and grain size requirements. This constitutes three separate grades. It is more economical if the mills can melt steel to only two, not three, different levels of carbon, and dual certify. Consider 304, UNS S30400. As the carbon is specified only as a maximum, it might be possible to melt 304 to 0.03 max carbon. Lower carbon would also result in lower than the 30,000 psi yield strength required. However, using the AOD it is now possible to add a very small, precisely controlled amount of nitrogen. This does not harm intergranular corrosion resistance but it does tend to increase room temperature tensile properties. With care in annealing practice, it is possible to produce 304 with low enough carbon to meet the 304L specification, yet with high enough yield strength to meet 304 requirements. As this metal meets all specified requirements of both 304L and 304, the mill test report will show both S30403 and S30400, i.e., dual certified.

S30403/S30400 is appropriate for corrosion service but not for high-temperature mechanical properties. For useful creep rupture strength some minimum amount of carbon is required, typically 0.04%. The situation was addressed a few years ago by adding a number of H grades to ASTM A240, with controlled carbon for high-temperature strength. The stainless 304H, S30409, has carbon specified 0.04–0.10% for high-temperature strength. In addition, there are grain size and minimum anneal temperature requirements. The 304, S30400, has no requirement for minimum carbon, control of grain size, or annealing temperature. Therefore any 304H containing no more than 0.08% carbon will meet 304 requirements, and may be dual-certified with 304. One should note that dual certified 304L/304 is suited only for aqueous corrosion service but would have rather low strength at high temperature. Likewise dual-certified 304/304H is meant for high-temperature service but may be unsatisfactory for welded construction in a wet corrosive environment. In practice, there is rather little actual S30400 produced as sheet or plate at this time. Most is dual certified, one way or another.

Like carbon, sulfur can now readily be refined to very low levels, typically less than 0.005%. Compare this with typical ASTM A 240 levels of 0.030% S maximum. Usually, stainless intended for plate is refined to a low-sulfur level to improve hot workability. Plate is generally formed and welded, with rather little machining by the customer. Low sulfur is quite detrimental to machinability. As bar products are commonly meant to be machined, most stainless bar actually must be resulfurized to some level, about 0.02%, for improved machinability. When this plate is intended to be drilled for a tubesheet, machineability becomes important and a resulfurized grade, still within the old 0.030 sulfur maximum, may be chosen.

It is precise control of chemistry, in particular nitrogen, that has permitted development of the superaustenitic 6% molybdenum grades. The ability to closely control nitrogen as an alloying addition has also tremendously improved the weldability of duplex stainless. Whereas formerly the only duplex stainless used in North America was 3RE60, and that only in tubing, today several grades of duplex stainless plate, pipe, and bar products are used in significant and growing amounts.

Chemistry control also means that the producer will minimize the use of expensive alloying elements. To remain competitive, mills now melt to the bottom end of the allowable range. One consequence for the user is that 316L stainless, specified as 2.00–3.00% molybdenum, now has a melt range of about 2.00–2.10% Mo. Through the 1970s the typical Mo level of 316L was about 2.3%. In addition, the average nickel content has dropped from just below 12% in the 1970s, down to around 10.2% Ni as currently produced. The result has occasionally been that 25-yr-old 316L equipment, replaced in kind, gave unexpectedly short life. In part, of course, this is due to increased corrosive conditions from recycling, rather than dumping, waste.

4 AVAILABILITY

The metallurgical aspects of alloy selection are well covered in this and other works. Along with corrosion resistance, strength, and fabricability, one of the most important material properties is availability. On a large project most alloys can be made in all product forms, in full mill heat lots. A contemporary example is 317LMX®, used as flat-rolled product, in mill heat lot quantities, for flue gas desulfurization scrubbers. This grade is rarely, if ever, carried in stock by mills or service centers.

Few mills produce all the forms, such as sheet, plate, bar, pipe, pipe fittings, and appropriate weld fillers, needed to fabricate a chemical process vessel. The minimum quantity a mill requires per product form is large, and lead times can be significant. This may limit flexibility in the event of error, last minute design changes, or later maintenance needs.

Consider what alternate, if more expensive, grade may be used to fill in for unavailable product forms or sizes. For example, AL-6XN has occasionally been used to fill out a bill of materials specified 317LMX®. The stainless grades 304L and 316L are used in such quantity that availability is unlikely to be an issue. The same may not be true for more highly alloyed or specialized materials.

5 FERRITIC STAINLESS STEEL

The ferritic grades listed below exhibit corrosion resistance over a very wide range. All are subject to severe embrittlement after prolonged exposure in the

700–1100°F (370–600°C) temperature range. Alloy 409 is weldable and is the alloy used for automotive exhaust systems and catalytic converter shells. It has some useful high-temperature oxidation resistance to about 1200°F (650°C). In plate gauges it may be welded with ERNiCr-3 for better toughness. Alloy 409 is not suited for decorative applications as it may rust from exposure to weather. Alloy 416 is free machining, available both as a ferritic and a martensitic version; 430 is commonly used for decorative purposes as well as food-handling equipment. E-BRITE has chloride pitting resistance equal or superior to 316L stainless, and practical immunity to chloride SCC. E-BRITE is currently available only as tubing for heat exchangers. AL29-4C is extremely resistant to chloride pitting and used for heat exchanger applications, available only as tubing or thin strip.

Ferritic Alloys (%)

Alloy	*UNS*	*Cr*	*Ni*	*Mo*	*Si*	*Mn*	*Cu*	*C*	*Fe*	*Other*
409	S40900	11	—	—	0.4	0.3	—	0.015	87	—
405	S40500	12	—	—	0.3	0.5	—	0.06	86	0.3Al
416	S41600	13	—	—	0.5	1	—	0.05	85	0.2S 0.04P
430	S43000	16.5	—	—	0.5	0.5	—	0.08	83	—
439	S43035	17.2	—	—	0.5	0.4	—	0.015	81	0.5Ti
E-BRITE	S44627	26	—	1	0.2	0.1	—	0.002	72	0.1Cb
AL29-4C	S44735	29	—	4	0.35	0.3	—	0.02	66	0.5Ti

6 MARTENSITIC STAINLESS STEELS

The only one of these that is readily welded is 410S. The higher carbon 410 may require significant preheat and postweld anneal. Consider the nickel alloy filler ERNiCr-3 for maximum resistance to weldment cracking, at a sacrifice in strength and hardness. The corrosion resistance of 410 depends upon the heat-treated condition. For maximum corrosion resistance temper either below 750°F (400°C) or above 1100°F (600°C). Both 410 and the martensitic 416 are used in sporting arms construction. The 440 series are used for cutting tools, 440A and B for pocket knives; 440C has the best edge retention and is used for fine custom knives and surgical tools.

Martensitic Alloys (%)

Alloy	*UNS*	*Cr*	*Ni*	*Mo*	*Si*	*Mn*	*C*	*Fe*	*Other*
410	S41000	12	—	—	0.3	0.4	0.14	87	—
410S	S41008	12	—	—	0.3	0.4	0.05	97	—
416	S41610	13	—	—	0.4	0.2	0.1	84	0.2S 0.05P
420	S42000	13	—	—	0.4	0.4	0.2	86	—
431	S43100	15.6	1.9	—	0.2	0.4	0.1	81	—
440A	S44002	17	—	0.5	0.2	0.4	0.67	81	—
440B	S44003	17	—	0.5	0.2	0.4	0.8	81	—
440C	S44004	17	—	0.5	0.2	0.4	1.07	80	—

7 AGE-HARDENING MARTENSITIC STAINLESS STEELS

These grades combine useful corrosion resistance with simple heat treatment. The most widely used is 17-4PH. They are susceptible to chloride stress–corrosion cracking. Susceptibility depends somewhat upon grade and aging temperature.

Age-Hardening Alloys (%)

Alloy	*UNS*	*Cr*	*Ni*	*Mo*	*Cu*	*Mn*	*Al*	*Ti*	*C*	*Fe*	*Other*
455	S45500	11.5	8.5	—	2	0.2	—	1.1	0.2	76	0.3Cb
PH15-7Mo®	S15700	15	7	2.5	—	0.4	1	—	0.04	74	—
17-4PH®	S17400	15.5	4.7	—	3.3	0.5	—	—	0.05	75	0.3Cb
17-7PH®	S17780	17	7.3	—	—	0.7	1.2	—	0.06	74	—
PH13-8Mo®	S13800	13	8	2.5	—	—	1.2	—	0.03	76	—

8 DUPLEX STAINLESS STEELS

Duplex stainless steels are characterized by high strength and good resistance to chloride stress–corrosion cracking. They represent a more economical solution to the problem of stainless SCC than the use of a high-nickel alloy. The lower grades, such as 2304, may be used to replace carbon steel in large part because the higher strength permits such weight reduction. The two grades most available from North American service centers, in a range of product forms, are 2205 and 2507. Alloys Zeron® 100, 2507, and 255 have sufficient chloride pitting resistance for seawater service.

Duplex Alloys (%)

Alloy	*UNS*	*Cr*	*Ni*	*Mo*	*Cu*	*Mn*	*Si*	*N*	*C*	*Fe*	*Other*
2304	S32304	23	4	0.3	0.3	2	—	0.1	0.03	70	—
3RE60	S31500	18.4	4.8	2.7	—	1.6	1.7	0.07	0.03	70	—
19D	S32001	21.2	1.3	—	—	5	0.4	0.15	0.02	71	—
2205	S31803	22	5.6	3.0	—	1.5	0.5	0.14	0.02	67	—
Zeron® 100	S32760	25	6.5	3.5	0.5	—	—	0.25	0.02	63	0.7W
2507	S32750	25	7	4	—	0.1	0.2	0.3	0.02	63	—
7-MoPLUS®	S32950	26.5	4.8	1.5	—	0.4	0.3	0.2	0.02	66	—
255	S32550	25.5	5.7	3.1	1.8	0.8	0.5	0.17	0.02	62	—

9 AUSTENITIC STAINLESS AND NICKEL ALLOYS

These nickel bearing alloys are used for the majority of corrosive environments. Alloy 316L is by far the most common choice for chemical process equipment. Formerly, when 316L was inadequate, the designer switched to the high end of the spectrum, alloy C-276, which is still considered the most broadly useful of the high alloys. Today there are numerous other choices, ranging from those of intermediate cost to alloys superior to C-276 in specific environments.

Types S30600, S32615, and alloy D-205™ are specialized high-silicon materials intended for concentrated sulfuric acid heat exchanger service. Weldability is limited. A very high chromium grade, R20033, is also intended for hot concentrated sulfuric acid. It is weldable and has a broader range of potential uses than do the high-silicon alloys.

Materials in the 5–7% molybdenum range are used for seawater service and chemical process vessels in general. Alloys N08367, S31254, and N08926 constitute the "6 moly" alloys. These have both corrosion resistance and cost intermediate between 316L and alloy C-276. All are readily fabricated and broadly available. Nitrogen in these alloys retards sigma formation, in spite of rather high Mo and moderate Ni levels. Prolonged exposure in the 1000–1800°F (540–980°C) temperature range may cause sigma-phase precipitation.

Alloys in the 13–16% Mo range include C-276, C-22, 59, C-2000, and 686. The latter four are regarded as improvements over C-276, with higher Cr for oxidizing environments. The newest, and possibly best, of the very high end nickel alloys, MAT 21, makes use of a tantalum addition, as well as 19% Mo.

Alloy selection for corrosive process environments is a complex process. It should include experience with similar equipment, extensive testing in the exact corrosive environment of interest, and detailed knowledge of the various alloys to be considered. Oftentimes, apparently minor contaminants can cause major changes in corrosion rates. One example is contamination of organic chlorides with small amounts of water. This can permit the organic compound to hydrolyze, forming hydrochloric acid. The HCl in turn may aggressively pit or stress corrode the standard 18–8 stainless steels. Other examples include the alloys B-2, 200, and 400, which contain no chromium. While they have excellent corrosion resistance in reducing environments, they have little or no resistance to oxidizing environments. Unexpected failures may therefore arise from contamination by small amounts of oxidizing salts (e.g., $FeCl_3$, $CuCl_2$, or $NaClO_3$), or even dissolved oxygen. Titanium behaves in the opposite manner and requires the presence of oxidizing species to develop its protective oxide film.

Austenitic Alloys (%)

Alloy	*UNS*	*Cr*	*Ni*	*Mo*	*Si*	*Mn*	*Cu*	*C*	*Fe*	*Other*
302	S30200	18.5	8.2	—	0.5	0.75	—	0.1	72	—
304L	S30403	18.3	9	—	0.5	1.7	—	0.02	70	—
321	S32100	17.3	9.3	—	0.7	1.8	—	0.01	70	0.2Ti
347	S34700	17	9.5	—	0.7	1.5	—	0.04	70	0.5Cb
316L	S31603	16.4	10.2	2.1	0.5	1.6	—	0.02	69	—
317L	S31703	18	11.6	3.1	0.4	1.5	—	0.02	65	—
317LMN	S31726	17	13	4.2	0.5	1.5	—	0.03	62	0.15N
A610	S30600	18	15	—	4	0.7	—	0.01	62	—
254 SMO	S31254	20	18	6.1	0.4	0.7	0.7	0.015	54	0.2N
SX	S32615	18	20.5	0.9	5.5	1.5	2	0.04	51	—
654 SMO®	S32654	24	22	7.3	—	3	0.5	0.01	42	0.5N
B66	S31266	24.5	22	5.6	—	3	1.5	0.02	44	2W
904L	N08904	21	25	4.5	0.5	1.7	1.6	0.015	45	—
1925hMO®, 25-6MO	N08926	20	25	6.2	0.4	0.7	0.9	0.01	46	0.2N
AL-6XN	N08367	20.5	24	6.3	0.4	0.3	—	0.02	48	0.22N

Alloy	*UNS*	*Cr*	*Ni*	*Mo*	*Si*	*Mn*	*Cu*	*C*	*Fe*	*Other*
28	N08028	27	31	3.5	0.2	1.8	1	0.01	35	—
31	N08031	27	31	6.5	0.2	1	1.2	0.01	33	0.2N
33	R20033	33	31	1.4	0.3	0.7	0.7	0.01	32	—
20Cb-3	N08020	20	33	2.2	0.4	0.4	3.3	0.02	40	0.5Cb
3620 Nb	N08020	20	37	2.1	0.3	1.6	3.4	0.02	35	0.6Cb
825	N08825	21.5	40	2.8	0.3	0.6	2	0.01	29	0.8Ti
RA333	N06333	25	45	3	1	1.5	—	0.05	18	3Wm3Co
G-30	N06030	29.5	45	5.5	—	—	1.9	0.01	15	0.7Cb 2.5W
G	N06007	22	45	6.5	0.3	1.5	2	0.01	20	2.1Cb 0.8W
G-3	N06985	22	48	7	0.4	0.8	2	0.01	18	0.3Cb 0.8W
625	N06625	21.5	61	9	0.1	0.1	—	0.05	4	3.6Cb
C-276	N10276	15.5	57	15.5	0.05	0.5	—	0.005	5.5	0.2V 4W
686	N06686	20.5	57	16.3	0.1	0.2	—	0.005	1	3.9W 0.2Al
C-2000®	N06200	23	58.5	16	0.02	0.2	1.6	0.003	0.3	0.25Al
C-22	N06022	21	57	13	0.05	0.3	—	0.003	4	0.2V 3W
59	N06059	23	59	16	0.05	0.4	—	0.005	0.3	0.2Al
MAT 21	—	19	60	19	—	—	—	—	—	1.8Ta
D-205	—	20	64	2.5	5	—	2	0.02	6	—
B-2	N10665	—	71	28	0.01	0.15	—	0.002	0.8	—
B	N10001	—	66	30	—	0.5	—	0.06	3	0.3V
B-3	N10675	1.3	67.7	28	0.03	0.5	—	0.002	1.5	—
B-4	N10629	1.3	67	28	—	—	—	0.005	3	—
400	N04400	—	66	—	0.02	1	31	0.1	1.5	—
201	N02201	—	99.5	—	0.2	—	—	0.01	0.2	—

10 WELDING[8]

There are some important distinctions between welding carbon steel and welding stainless steel. Alloys containing more than about 20% nickel have somewhat different requirements than the lower nickel austenitic stainless steels. There are specific issues regarding duplex stainless steels and weld fillers for the high-molybdenum grades.

10.1 Carbon Steel versus Stainless

Six important differences between welding the carbon or low-alloy structural steels and the austenitic stainless and nickel alloys are surface preparation, shielding gases, cold cracking versus hot cracking, distortion, penetration, and fabrication time.

1. *Surface Preparation.* When fabricating carbon steel, it is common practice to weld right over the scale (a so-called mill finish is a layer of blue-black oxide, or scale, on the metal surface), red rust, and even paint. The steel weld fillers normally contain sufficient deoxidizing agents, such as manganese and silicon, to reduce these surface iron oxides back to metallic iron. The resultant Mn–Si slag floats to the weld surface. Iron oxide, or scale, melts at a lower temperature, 2500°F (1371°C), than does the steel itself. One can see this in a steel mill when a large ingot is removed from the soaking pit for forging—the molten scale literally drips off the white-hot steel.

Stainless steel, by contrast, must be clean and free of any black scale from hot rolling, forging, or annealing operations. Such scale is predominately chromium oxide. Normally, stainless is produced with a descaled white or bright finish. Stainless steel melts at a lower temperature than does its chromium oxide scale, and the stainless weld filler chemistry is not capable of reducing this scale back to metallic chromium. As a result, with gas-shielded processes it is difficult to get the weld bead to even "wet," or stick to, a scaled piece of stainless.

The need to clean or grind down to bright metal is more likely to cause trouble when stainless is being joined to carbon steel. In this dissimilar metal joint it is necessary to grind that carbon steel to bright metal, on both sides of the joint, free of all rust, mill scale, grease, and paint. The preferred weld fillers for this particular joint, to minimize the hard martensitic layer on the steel side, are ENiCrFe-3-covered electrodes or ERNiCr-3 bare filler wire; E309 electrodes are commonly used but are not the optimum choice.

Both stainless and high-nickel alloys designed for corrosion resistance are produced to very low carbon contents, less than 0.03% and sometimes less than 0.01% carbon. Any higher carbon will reduce the metal's corrosion resistance. For this reason it is necessary to clean the alloy thoroughly of all grease and oil before welding. Also the very high nickel alloys, such as Monel 400 or commercially pure nickel 200/201, are very sensitive to weld cracking from the sulfur in oil or marking crayons.

Metallic zinc paint is a common way to protect structural steel from corrosion. Even a small amount of that zinc paint overspray on stainless will cause the stainless to crack badly when welded. Consider finishing all stainless welding before painting the structural steel in the area.

2. *Shielding Gases.* For gas–metal arc welding (GMAW, a.k.a. MIG) carbon steel the shielding gases are usually 95% argon 5% oxygen, 75% argon 25% carbon dioxide, or 100% CO_2. While suitable for carbon or low-alloy steel welding wire, such gases are far too oxidizing for use with stainless or nickel alloys. It is not unknown to hear the complaint "clouds of red smoke are coming off when I weld this stainless . . . heavy spatter" and then learn that the shielding gas used was 75% argon 25% CO_2. A fine gas for carbon steel but not for any stainless.

One exception to this high CO_2 prohibition is when using flux-cored wire, either stainless or nickel alloy. Some of these wires are specifically formulated to run best with 75% Ar 25% CO_2.

Stainless and nickel alloys may be GMAW spray-arc welded using 100% argon shielding gas, though it is not necessarily the ideal choice. Stainless steels possess a passive chromium oxide film. The basis of stainless corrosion resistance, this film is not desirable when welding. Some 80% argon is necessary to get into a spray-transfer mode. Beyond this, a helium addition, up to about 20%, gives a hotter arc and helps break up the oxide film. A very slight amount of carbon dioxide, perhaps 1–2%, will prevent arc wander. Too much CO_2 may begin to add carbon to the weld, undesirable for corrosion resistance. Nitrogen may be added in gases specifically designed for welding the duplex stainless steels.

Pulse-arc welding is usually done with a 75% argon 25% helium mix. Short-circuiting arc transfer may be done with this 75% Ar–25% He mix but a 90% helium $7\frac{1}{2}$% argon $2\frac{1}{2}$% CO_2 "tri-mix" is more commonly used.

3. *Cold Cracking versus Hot Cracking.* Carbon steel weldments may harden, and crack, as they cool from welding. High hardness, and the resulting cracking, may occur when the steel contains more than 0.25% carbon. Alloying elements that increase hardenability, such as manganese, chromium, molybdenum, etc., can make steels of lower carbon content also harden. Hydrogen pickup from moisture in the air causes underbead cracking in steels that harden as they cool from welding. To prevent such cracking, the steel is usually preheated before welding. This retards the cooling rate of the weld and avoids martensite formation. Postweld heat treatment, or stress relief, is also applied to some steels, or for certain applications. The martensitic stainless steels behave in this fashion and because of their high hardenability are quite difficult to weld.

Austenitic stainless and nickel alloys do NOT harden, no matter how fast they cool from welding. So, it is not necessary to preheat austenitic stainless, nor to postweld heat treatment. Indeed preheating austenitic alloys, beyond what may be necessary to dry the metal, can be positively harmful. Stress relief 1100–1200°F (600–650°C) as applied to carbon steel is ineffective with stainless or nickel alloys and may damage the corrosion resistance of some grades.

Stainless steel weldments are usually quite resistant to cracking, unless contaminated, possibly by zinc or copper, more rarely by aluminum. A small amount of ferrite in the austenitic weld bead provides this hot cracking resistance.

High-nickel alloys are less forgiving and may be susceptible to cracking in restrained joints, including heavy sections. This is a hot tearing, not a cold crack. That is, the weld bead tears rather than stretching, as the bead contracts upon solidifying.

This hot tearing/hot cracking has nothing to do with hardness. The faster a nickel alloy weld freezes solid, the less time it spends in the temperature range where it can tear. For this reason preheating, which slows down the cooling rate, is actually harmful, as it permits more opportunity for hot tearing to occur.

4. *Distortion.* Stainless steel has poor thermal conductivity, only about one fourth that of ASTM A36 structural steel. This means the welding heat tends to remain concentrated, rather than spread out. Stainless also expands with heat about half again as much as does carbon steel. The combination of these two factors means that stainless or nickel alloy fabrications distort significantly more than similar designs in carbon steel.

Tack welds should be closer than with carbon steel and sequenced in a pattern, left side, right side, middle, etc. If the tacks are simply done in order from one end, the plate edges close up. To balance stresses, weld runs should be done symmetrically about the joint's center of gravity. Back step welding is helpful. This subject is well covered in Refs. 9 and 10.

Reducing heat input reduces the stresses and distortions from the welding operation. Heat into the workpiece is controlled by welding current, arc voltage, travel speed, and the specific welding process used. For the same amperes, volts and speed, submerged arc welding (SAW) transfers the most heat; shielded metal arc (SMAW), and gas–metal arc (GMAW) with argon shielding next and roughly equivalent. Gas–tungsten arc welding (GTAW) can put the least heat into the work.

5. *Penetration.* There is a tendency to increase heat input with the stainless and high-nickel alloys. First, the weld fillers tend to be sluggish and not flow well, as compared with carbon steel. Second, the arc simply will not penetrate stainless steel as it does carbon steel. The higher the nickel, the less the penetration. Increasing welding current will not solve the problem, and may cause cracking in the higher nickel grades. Stainless, and especially nickel alloy, joint designs must be more open. The base metal should be single or double beveled, with a root gap, so that the weld metal may be placed in the joint.

6. *Fabrication Time.* Cleanliness, distortion control measures, maintaining low interpass temperatures, and even machining add up to more time spent fabricating stainless than carbon steel. A shop experienced with stainless may require 1.6 times as long to complete the same fabrication in stainless, as in carbon steel. A good carbon steel shop encountering stainless or nickel alloys for the first time may easily spend twice as long, maybe even three times as long, to do the stainless fabrication, as it would the same job in carbon steel.

10.2 Austenitic Alloys

The fundamental problem to be overcome in welding austenitic nickel bearing alloys is the tendency of the weld to hot tear upon solidification from the melt. This matter is readily handled in stainless steels of up to about 15% or so nickel. In these stainless grades the weld metal composition is adjusted, usually by slightly higher chromium and reduced nickel, to form a small amount of ferrite upon solidification. The amount of ferrite in the weld may be measured magnetically, and is reported as a ferrite number (FN). This ferrite acts to nullify the effects of the elements responsible for hot cracking in the Ni–Cr–Fe austenitics. These elements are chiefly phosphorus, sulfur, silicon, and boron. In higher nickel grades, about 20% nickel and over, it is metallurgically not possible to form any measurable amount of ferrite. Therefore other means of minimizing hot cracking must be used. Foremost among these is to use high purity raw materials in the manufacture of weld fillers. Simply reducing the amounts of harmful P, S, Si, and B in the weld metal improves its ability to make a sound weld. Phosphorus, in particular, must be kept below 0.015% in the weld wire itself. Certain alloy additions such as manganese, columbium (niobium), and molybdenum serve in one way or another to reduce the austenitic propensity for weld hot cracking. Manganese ranges from about 2% in AWS E310-15-covered electrodes to 8% in ENiCrFe-3-nickel-alloy-covered electrodes. Columbium at the 0.5% level, as in 347 or 20Cb-3 stainless, is harmful whereas 2–4% Cb is quite beneficial in many nickel base weld fillers. Molybdenum is not added with the intention of increasing weldability, nevertheless it does so. The 2% Mo contributes to 316L being the most weldable of the stainless steels, and 15% in C-276 accounts for the popularity of the various "C-type" electrodes in repair welding.

The distinction between the lower nickel stainless grades, which depend upon ferrite to ensure weldability, and the high-nickel alloys, which require high purity weld fillers, is an important one to remember. Most ferrite-containing (stainless) weld fillers are useless with nickel alloy base metal, as dilution of the weld bead with nickel from the base metal eliminates this ferrite. Likewise a high purity nickel alloy weld filler, such as ER320LR, may not be quite so crack resistant when contaminated by phosphorus from use on 316L or carbon steel base metal.

Alloys under 20% Nickel

Most austenitic grades containing less than 20% nickel are joined with weld fillers that utilize some ferrite, perhaps 4-12 FN, to ensure freedom from hot cracks. In practice, this means stainless steels up through 317LMN or 309S, both about 13% nickel. The 310S type stands in an odd position between the stainless and the nickel alloys, having neither ferrite nor any particular alloy addition to the weld metal to ensure sound welds. Not surprisingly, 310S welds have a reputation for fissuring.

Alloys over 20% Nickel

Corrosion-resistant alloys in this category begin with the 18% nickel 254 SMO, for this and other "6 moly" grades are welded with an overmatching nickel alloy filler such as ERNiCrMo-3.

Other nickel alloys are joined with matching composition weld fillers modified only by restrictions on P, S, Si, and B. A minor amount of titanium may be added for deoxidation. Other fillers contain significant manganese or columbium to improve resistance to fissuring and hot cracking.

Such chemistry modifications are rarely as effective as is the use of ferrite in the lower nickel stainless weld fillers. Welding technique and attention to cleanliness, then, become increasingly important to ensure the soundness of fully austenitic welds. These techniques include bead contour and low interpass temperature. Reinforced, convex stringer beads are much more resistant to centerbead cracking than are shallow, concave beads. Interpass temperature for most nickel alloy weldments is kept below 300°F (150°C). Cleanliness includes NOT using oxygen additions to the GMAW shielding gases for nickel alloys.

It is worth repeating here that high-nickel alloys cannot be welded using stainless steel weld fillers. Stainless steel fillers (308, 309, etc.) depend upon a chemistry that will solidify from the melt as a duplex structure, containing a small amount of ferrite in with the austenite.

10.3 Duplex Stainless Steels[11]

When welding duplex stainless the issues are maintaining the austenite–ferrite balance in the weldment and avoiding precipitation of nitrides and sigma. Rather high heat input is used to weld duplex, similar to welding 316L stainless. This is in contrast to the lower heat preferred with the high-nickel alloys. Low heat input is positively NOT desirable with a duplex, as it may not permit sufficient transformation of ferrite to austenite upon cooling. Current duplex weld fillers have nickel content somewhat increased over that of the base metal. This is to ensure that enough weld metal ferrite transforms to austenite, to maintain a balance of the two phases.

Toughness and critical pitting temperature of duplex weldments vary with the choice of welding process, in opposite directions. In order of increasing notch impact toughness: SMAW AC/DC, FCAW (flux cored arc welding), SMAW

DC-basic, SAW, GMAW argon shielding, GMAW 95AR 3He $2N_2$ shielding, and GTAW. In order of increasing pitting resistance, as measured by critical pitting temperature: GTAW, GMAW, SAW, FCAW, SMAW DC-bsic, and SMAW AC/DC.

10.4 High-Molybdenum Alloys[12]

High-molybdenum-containing stainless and nickel alloys are welded with an overmatching filler metal. This is necessary to maintain corrosion resistance in the weld metal at least equal to the base metal. The reason is that molybdenum and chromium segregate as the weld metal solidifies from the melt. This leaves local areas with high, and with low, molybdenum. Pitting corrosion can start in the low-Mo area, with the pits eventually growing even into metal with high molybdenum. This occurs in alloys ranging from 316L to C-276, being more severe at higher alloy contents.

This matter began to receive attention when the 6% molybdenum stainless steels came on the market. If any of these 6% Mo grades are welded without filler metal, the result is a weld bead that may be as low as 3% Mo in areas. The end result can be that this weld has only the pitting corrosion resistance of 317L stainless. In the case of tubular products autogenously welded in production, a high-temperature anneal is used to homogenize the metal. In addition, a small amount of nitrogen, 3–5%, is added to the torch gas. Fabrications of thin sheet, which cannot be annealed after welding, should have this nitrogen addition to minimize the loss of corrosion. In addition, thin sheet welds solidify more quickly, hence the segregation is less severe.

In normal fabrication of a 6% molybdenum grade, alloy 625 (ERNiCrMo-3) filler metal is used. The weld metal contains 9% Mo. After welding, segregation causes some areas to have as little as 6% molybdenum. The result is that the alloy 625 weld bead has approximately the same corrosion resistance as the 6% molybdenum base metal. Higher alloy weld fillers, such as ERNiCrMo-10 or ERNiCrMo-14, may also be used, though the benefit may be more theoretical than real. ERNiCrMo-4 is not suggested, as it has 5% less chromium than does AL-6XN, for example. Since the mid-1980s nearly all of the 6% molybdenum alloy fabrications have been made, and put into service, using a 9% molybdenum weld filler.

ERNiCrMo-3 weld filler is widely available and is appropriate for welding lower alloys such as 317L, 317LMN, and 904L for chloride service. This molybdenum, and chromium, segregation problem exists with the 13–16% Mo nickel alloys as well. In this case there is not much choice in higher alloy weld fillers.

WEB SITES

Detailed technical information on heat and corrosion resistant alloys is available at www.rolledalloys.com. Complete stainless steel data is at www.alleghenyludlum.com. Nickel alloys in general are covered at www.nidi.org.

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CHAPTER 4

ALUMINUM ALLOYS

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1 NATURE OF ALUMINUM ALLOYS

Aluminum alloys are broadly used in products and applications that touch us regularly in our daily lives, from aluminum foil for food packaging and easy-open aluminum cans for beverages to the structural members of the aircraft in which we travel. The broad use of aluminum alloys is dictated by a very desirable combination of properties, combined with the ease with which they may be produced in a great variety of forms and shapes.

The first step in becoming familiar with the opportunities to utilize aluminum alloys advantageously is to briefly note some of the basic characteristics of wrought aluminum alloys that make them desirable candidates for such a wide range of applications, as well as their limitations. Wrought alloys (those mechanically formed by rolling, forging, and extrusion into useful products) are addressed first, then cast alloys (those cast directly to the near-final finished shape).

For readers who are interested in a broader look at the aluminum industry, the publications of the Aluminum Association, Inc., are highly recommended, especially:

- D. G. Altenpohl, *Aluminum: Technology, Applications, and Environment,* The Aluminum Association, Washington, DC, and TMS, Warrendale, PA 1998

Handbook of Materials Selection. Edited by Myer Kutz

- *Aluminum Standards & Data* (*Standard and Metric Editions*), The Aluminum Association, Inc., Washington, DC, published periodically
- *The Aluminum Design Manual,* The Aluminum Association, Inc., Washington, DC, 2000.

See the list of references at the end of the chapter for a more complete listing.

1.1 Advantages of Wrought Aluminum Alloys

1. *Corrosion Resistance.* As a result of a naturally occurring tenacious surface oxide film, many aluminum alloys provide exceptional resistance to corrosion in many atmosphere and chemical environments. Alloys of the 1xxx, 3xxx, 5xxx, and 6xxx systems are especially favorable in this respect and are even used in applications where they are in direct contact with seawater and antiskid salts. With some electrocoating enhancements (e.g., anodizing), the oxide coating can be thickened for even greater protection.

2. *Thermal Conductivity.* Aluminum and its alloys are good conductors of heat, and, while they melt at lower temperatures than steels, about 1000°F (about 535°C), they are slower to reach very high temperatures than steel in fire exposure.

3. *Electrical Conductivity.* Pure aluminum and some of its alloys have exceptionally high electric conductivity (i.e., very low electrical resistivity), second only to copper among common metals as conductors.

4. *Strength/Weight Ratio.* The combination of relatively high strength with low density means a high strength efficiency for aluminum alloys and many opportunities for replacement of heavier metals with no loss (and perhaps a gain) in load-carrying capacity. This characteristic, combined with the excellent corrosion resistance and recyclability, has led to aluminum's broad use in containers, aircraft, and automotive applications.

5. *Fracture Toughness and Energy Absorption Capacity.* Many aluminum alloys are exceptionally tough and excellent choices for critical applications where resistance to brittle fracture and unstable crack growth are imperatives. Alloys of the 5xxx series, for example, are prime choices for liquified natural gas (LNG) tankage. And special high-toughness versions of aircraft alloys, such as 2124, 7050, and 7475, replace the standard versions of these alloys for critical bulkhead applications.

6. *Cryogenic Toughness.* Aluminum alloys, especially of the 3xxx, 5xxx, and 6xxx series, are ideal for very low temperature applications because the ductility and toughness as well as strength of many alloys at subzero temperatures are as high as or higher than at room temperature, even down to near absolute zero.

7. *Fatigue Strength.* On an efficiency basis (strength to density) the fatigue strengths of many aluminum alloys are comparable to those of steels.

8. *Modulus of Elasticity.* Aluminum alloys have elastic moduli about one third those of steels (about 10,000,000 psi vs. about 30,000,000 psi), so they absorb about three times as much elastic energy upon deformation to the same stress. They also deflect three times more under load (see Section 1.3).

9. *Workability.* Aluminum alloys are readily workable by a great variety of metal-working technologies and especially amenable to extrusion (the process of forcing heated metal through shaped dies to produce specific shaped sections). This enables aluminum to be produced in a remarkable variety of shapes and forms in which the metal can be placed in locations where it can most efficiently carry the applied loads.

10. *Ease of Joining.* Aluminum alloys may be joined by a very broad variety of commercial methods including welding, brazing, soldering, riveting, bolting and even nailing in addition to an unlimited variety of mechanical procedures. Welding, while considered difficult by those familiar only with joining steel and who try to apply the same techniques to aluminum, is particularly easy when performed by proven techniques such as gas–metal arc welding (GMAW or MIG) or gas–tungsten arc welding (GTAW).

11. *Recyclability.* Aluminum and its alloys are among the easiest to recycle of any structural materials. And they are recyclable in the truest sense, unlike materials that are reused but in lower quality products: Aluminum alloys may be recycled *directly* back into the same high-quality products like rigid container sheet (cans) and automotive components.

1.2 Advantages of Cast Aluminum Alloys

The desirable characteristics of wrought alloys are also generally applicable to cast alloys, but in fact the choice of one casting alloy or another tend to be more often made on the basis of their relative abilities to meet one or more of the following characteristics:

Ease of Casting. Many aluminum casting alloys have relatively high-silicon contents that provide excellent flow characteristics during casting, enabling them to be utilized for large and complex castings (even complete engines, for example). Relatively minute details in the shape of the casting can be accurately and reliably replicated.

High Strength. Many aluminum casting alloys respond to heat treatment following casting and achieve relatively high levels of strength and excellent strength/weight ratios. With careful design of molds, high chill rates can be assured and both high strength and high toughness can be achieved.

Quality of Finish. By proper selection of aluminum casting alloy, extremely fine surface quality can be achieved. While such alloys typically require more attention to casting practice typically, they are widely used in applications where the finished casting surface mirrors the finish needed in surface.

Unfortunately few casting alloys possess all three characteristics, but some generalizations may be made:

1. The high-silicon 3xx.x series are outstanding with respect to ease of casting as the relatively high silicon contents lend a characteristic of good flow and mold filling capability. As a result, the 3xx.x series are the most widely used and especially chosen for large and very complex casting.
2. The 2xx.x alloys typically provide the very highest strengths but are more difficult to cast and lack good surface characteristics. Therefore their use

is usually limited to situations where expert casting techniques can be applied and where strength and toughness are at a premium, as in the aerospace industry.

3. The 5xx.x and 7xx.x series are noteworthy for the fine finish they provide, but they are more difficult to cast than the 3xx.x series and so are usually limited to those applications where that finish is paramount. A good example is the use of 7xx.x alloys for bearings.

1.3 Limitations of Wrought and Cast Aluminum Alloys

There are several characteristics of aluminum alloys that require special attention in alloy selection or design:

1. *Moduli of Elasticity.* As noted earlier, the elastic moduli of aluminum alloys are about one third those of steel. In applications such as bridges, where some designs may be deflection critical, consideration should be given to the fact that aluminum alloys will deflect about three times more than comparably sized steel members; in order to compensate for this aluminum members subject to bending loads are usually made deeper or thicker in their upper and lower extremities to reduce stresses and/or deflections.

2. *Melting Temperature.* Aluminum alloys melt at about 1000°F (535°C), well below where steels melt, and so they should not be selected for applications such as flue pipes and fire doors where the low melting point may result in unsatisfactory performance. The useful limit of high-temperature structural application of aluminum alloys is about 500°F (about 260°C) for conventional alloys or about 600°F (315°C) for alumina-enhanced alloys. It is useful to note that even in the most intense fires, aluminum alloys do not burn; they are rated noncombustible in all types of fire tests and achieve the highest ratings in flame-spread tests. Further, as noted earlier, they are slower to reach high temperatures in fires than other metals such as steels because of their higher thermal conductivity and emmissivity. Nevertheless, they should not be used where service requirements include structural strength above 500°F or exposure above 1000°F.

3. *Stress–Corrosion Susceptibility of Some Alloys.* Some aluminum alloys, notably the 2xxx and 7xxx alloys, when stressed perpendicular to the major plane of grain flow (i.e., in the short-transverse direction) may be subject to intergranular stress–corrosion cracking (SCC) unless they have been given a special thermal treatment to reduce or eliminate this type of behavior. If short transverse stresses are anticipated in relatively thick components, 2xxx alloys should only be utilized in the T6- or T8-type tempers (not T3- or T4-type tempers), and 7xxx alloys should only be used in the T73-type tempers (not T6- or T8-type tempers). Similarly, 5xxx alloys with more than 3% Mg should not be used in applications where a combination of high stress and high temperature will be experienced over a long period of time (more than several hundred hours), because some susceptibility to SCC may be encountered (i.e., the alloys may become "sensitized"); for applications where temperatures above about 150°F are likely to be encountered for long periods, the use of 5xxx alloys with 3% or less Mg is recommended.

4. *Mercury Embrittlement.* Aluminum alloys should never be used when they may be in direct contact with liquid or vaporized mercury; severe grain-boundary embrittlement may result. This is an unlikely exposure for the vast majority of applications, but in any instance where there is the possibility of mercury being present, the use of aluminum alloys should be avoided.

2 DESIGNATION SYSTEMS

One advantage in using aluminum alloys and tempers is the universally accepted and easily understood alloy and temper systems by which they are known. It is extremely useful for both secondary fabricators and users of aluminum products and components to have a working knowledge of the those designation systems. The alloy system provides a standard form of alloy identification that enables the user to understand a great deal about the chemical composition and characteristics of the alloy, and similarly, the temper designation system permits one to understand a great deal about the way in which the product has been fabricated.

The alloy and temper designation systems in use today for wrought aluminum were adopted by the aluminum industry in about 1955, and the current system for cast system was developed somewhat later. The aluminum industry managed the creation and continues the maintenance of the systems through its industry organization, The Aluminum Association. The alloy registration process is carefully controlled and its integrity maintained by the Technical Committee on Product Standards of the Aluminum Association, made up of industry standards experts. Further, as noted earlier, the Aluminum Association designation system is the basis of the American National Standards Institute (ANSI) standards, incorporated in ANSI H35.1[1] and, for the wrought alloy system at least, is the basis of the near-worldwide International Accord on Alloy Designations.[2]

The Aluminum Association Alloy and Temper Designation Systems covered in ANSI H35.1 and aluminum standards and data[3] are outlined in this chapter.

2.1 Wrought Aluminum Alloy Designation System

The Aluminum Association Wrought Alloy Designation System consists of four numerical digits, sometimes with alphabetic prefixes or suffices, but normally just the *four* numbers:

- The *first* digit defines the major alloying class of the series, starting with that number.
- The *second* defines variations in the original basic alloy; that digit is always a zero (0) for the original composition, a one (1) for the first variation, a two (2) for the second variation, and so forth; variations are typically defined by differences in one or more alloying elements of 0.15–0.50% or more, depending upon the level of the added element.
- The *third and fourth* digits designate the specific alloy within the series; there is no special significance to the values of those digits except in the 1xxx series (see below), nor are they necessarily used in sequence.

Table 1 shows the meaning of the first of the four digits in the alloy designation system. The alloy family is identified by that number and the associated main alloying ingredient(s), with three exceptions:

Table 1 Main Alloying Elements in Wrought Alloy Designation System

Alloy	Main Alloying Element
1000	Mostly pure aluminum; no major alloying additions
2000	Copper
3000	Manganese
4000	Silicon
5000	Magnesium
6000	Magnesium and silicon
7000	Zinc
8000	Other elements (e.g., iron or tin)
9000	Unassigned

- Members of the 1000 series family are commercially pure aluminum or special purity versions, and as such do not typically have any alloying elements intentionally added; however they do contain minor impurities that are not removed unless the intended application requires it.
- The 8000 series family is an ‘‘other elements’’ series, comprised of alloys with rather unusual major alloying elements such as iron and nickel.
- The 9000 series is unassigned.

The major benefits of understanding this designation system are that one can tell a great deal about the alloy just from knowing to which series it belongs.

- As indicated earlier, the 1xxx series are pure aluminum and its variations; compositions of 99.0% or more aluminum are by definition in this series. Within the 1xxx series, the last two of the four digits in the designation indicate the minimum aluminum percentage. These digits are the same as the two digits to the right of the decimal point in the minimum aluminum percentage specified for the designation when expressed to the nearest 0.01%. As with the rest of the alloy series, the second digit indicates modifications in impurity limits or intentionally added elements. Compositions of the 1xxx series do not respond to any solution heat treatment but may be strengthened modestly by stain hardening.
- The 2xxx series alloys have copper as their main alloying element and, because it will go in significant amounts into solid solution in aluminum, they will respond to solution heat treatment; they are referred to as heat treatable.
- The 3xxx series alloys are based on manganese and are strain hardenable; they do not respond to solution heat treatment.
- The 4xxx series alloys are based on silicon; some alloys are heat treatable, others are not, dependent upon the amount of silicon and the other alloying constituents.
- The 5xxx series alloys are based on magnesium and are strain hardenable, not heat treatable.

- The 6xxx series alloys have both magnesium and silicon as their main alloying elements; these combine as magnesium silicide (Mg_2Si) following solid solution, and so the alloys are heat treatable.
- The 7xxx series alloys have zinc as their main alloying element, often with significant amounts of copper and magnesium, and they are heat treatable.
- The 8xxx series contain one or more of several less frequently used major alloying elements like iron or tin; their characteristics depend on the major alloying element(s).

The compositions of a representative group of widely used commercial wrought aluminum alloys are given in Table 2, taken from *Aluminum Standards & Data*[3] and other Aluminum Association publications.

2.2 Cast Aluminum Alloy Designation System

The designation system for cast aluminum alloys is similar in some respects to that for wrought alloys but has a few very important differences as noted by the following description.

Like the wrought alloy system, the cast alloy designation system also has four digits, but differs from the wrought alloy system in that a decimal point is used between the third and fourth digits to make clear that these are designations to identify alloys in the form of castings or foundry ingot.

As for the wrought alloy designation system, the various digits of the cast alloy system convey information about the alloy:

- The first digit indicates the alloy group, as can be seen in Table 3. For 2xx.x through 8xx.x alloys, the alloy group is determined by the alloying element present in the greatest mean percentage, except in cases in which the composition being registered qualifies as a modification of a previously registered alloy. Note that in Table 3, the 6xx.x series is shown last and for cast alloys is designated as the unused series.
- The second and third digits identify the specific aluminum alloy or, for the aluminum 1xx.x series, indicate purity. If the greatest mean percentage is common to more than one alloying element, the alloy group is determined by the element that comes first in sequence. For the 1xx.x group, the second two of the four digits in the designation indicate the minimum aluminum percentage. These digits are the same as the two digits to the right of the decimal point in the minimum aluminum percentage when expressed to the nearest 0.01%.
- The fourth digit indicates the product form: xxx.0 indicates castings, and xxx.1 for the most part indicates ingot having limits for alloying elements the same as those for the alloy in the form of castings. A fourth digit of xxx.2 may be used to indicate that the ingot has composition limits that differ from but fall within the xxx.1 limits; this typically represents the use of tighter limits on certain impurities to achieve specific properties in the cast product produced from that ingot.

Table 2 Nominal Compositions of Wrought Aluminum Alloy[a]

	Percent of Alloying Elements[b]								
Alloy	Silicon	Copper	Manganese	Magnesium	Chromium	Nickel	Zinc	Titanium	Notes[c]
1060	—	—	—	—	—	—	—	—	1
1100	—	—	—	—	—	—	—	—	1
1145	—	—	—	—	—	—	—	—	1
1350	—	—	—	—	—	—	—	—	2
2008	0.65	0.9	—	0.38	—	—	—	—	3
2010	—	1.0	0.25	0.7	—	—	—	—	
2011	—	5.5	—	—	—	—	—	—	4
2014	0.8	4.4	0.8	0.50	—	—	—	—	
2017	0.50	4.0	0.7	0.6	—	—	—	—	
2024	—	4.4	0.6	1.5	—		—	—	
2025	0.8	4.4	0.8		—		—	—	
2036	—	2.6	0.25	0.45	—	—	—	—	
2117	—	2.6	—	0.35	—	—	—	—	
2124	—	4.4	0.6	1.5	—	—	—	—	5
2195	—	4.0	—	0.50	—	—	—	—	5
2219	—	6.3	0.30	—	—	—	—	0.06	7
2319	—	6.3	0.30	—	—	—	—	0.15	7
2618	0.18	2.3	—	1.6	—	1	—	0.07	8
3003	—	.12	1.2	—	—	—	—	—	
3004	—	—	1.2	1	—	—	—	—	
3005	—	—	1.2	0.40	—	—	—	—	
3105	—	—	0.6	0.50	—	—	—	—	
4032	12.2	0.9	—	—	—	—	0.9	—	
4043	5.2	—	—	—	—	—	—	—	
4643	4.1	—	—	0.20	—	—	—	—	
5005	—	—	—	0.8	—	—	—	—	
5050	—	—	—	1.4	—	—	—	—	
5052	—	—	—	2.5	0.25	—	—	—	
5056	—	—	0.12	5.0	0.12	—	—	—	
5083	—	—	0.7	4.4	0.15	—	—	—	
5086	—	—	0.45	4.0	0.15	—	—	—	
5154	—	—	—	3.5	0.25	—	—	—	
5183	—	—	0.8	4.8	0.15	—	—	—	
5356	—	—	0.12	5.0	0.12	—	—	0.13	
5454	—	—	0.8	2.7	0.12	—	—	—	
5456	—	—	0.8	5.1	0.12	—	—	—	
5457	—	—	0.30	1.0	—	—	—	—	
5554	—	—	0.8	2.7	0.12	—	—	0.12	
5556	—	—	0.8	5.1	0.12	—	—	0.12	
5657	—	—	—	0.8	—	—	—	—	
5754	—	—	0.5	3.1	0.3	—	—	—	
6005	0.8	—	—	0.50	—	—	—	—	
6009	—	—	—	—	—	—	—	—	
6013	0.8	0.8	0.50	1.0	—	—	—	—	
6053	0.7	—	—	1.2	0.25	—	—	—	
6061	0.6	0.28	—	1.0	0.20	—	—	—	
6063	0.40	—	—	0.7	—	—	—	—	
6066	1.4	1.0	0.8	1.1	—	—	—	—	
6070	1.4	0.28	0.7	0.8	—	—	—	—	
6101	0.50	—	—	0.6	—	—	—	—	
6111	0.85	0.7	0.28	0.75	—	—	—	—	
6151	0.9	—	—	0.6	0.25	—	—	—	
6201	0.7	—	—	0.8	—	—	—	—	
6262	0.6	0.28		1.0	0.09				9
6351	1.0	—	0.6	0.6	—	—	—	—	
6951	0.35	0.28	—	0.6	—	—	—	—	

Table 2 (*Continued*)

	Percent of Alloying Elements[b]								
Alloy	Silicon	Copper	Manganese	Magnesium	Chromium	Nickel	Zinc	Titanium	Notes[c]
7005	—	—	0.45	1.4	0.13	—	4.5	0.04	10
7049	—	1.6	—	2.4	0.16	—	7.7	—	
7050	—	2.3	—	2.2	—	—	6.2	—	11
7072	—	—	—	—	—	—	1.0	—	
7075	—	1.6	—	2.5	0.23	—	5.6	—	
7116	—	0.8	—	1.1	—	—	4.7	—	12
7129	—	0.7	—	1.6	—	—	4.7	—	12
7175	—	1.6	—	2.5	0.23	—	5.6	—	5
7178	—	2.0	—	2.8	0.23	—	6.8	—	
7475	—	1.6	—	2.2	0.22	—	5.7	—	5
8017	—	0.15	—	0.03	—	—	—	—	13
8090	—	1.3	—	0.95	—	—	—	—	14
8176	0.09	—	—	—	—	—	—	—	13

[a] Based upon industry handbooks.[3,4] Consult those references for specified limits. Values are nominal, i.e., middle range of limits for elements for which a composition range is specified.

[b] Aluminum and normal impurities constitute remainder.

[c]
1. Percent minimum aluminum—for 1060: 99.60%; for 1100: 99.00%; for 1145: 99.45%; for 1350: 99.50%. Also, for 1100, 0.12% iron.
2. Formerly designated EC.
3. Also contains 0.05% vanadium (max.).
4. Also contains 0.40% lead and 0.4% bismuth.
5. This alloy has tighter limits on impurities than does its companion alloy (2024 or 7075).
6. Also contains 1.0% lithium, 0.42% silver, and 0.12% zirconium.
7. Also contains 0.10% vanadium plus 0.18% zirconium.
8. Also contains 1.1% iron.
9. Also contains 0.55% lead and 0.55% bismuth.
10. Also contains 0.14% zirconium.
11. Also contains 0.12% zirconium.
12. Also contains 0.05% max. vanadium plus 0.03% max. gallium.
13. Also contains 0.7% iron.
14. Also contains 2.4% lithium plus 0.10% zirconium.

Table 3 Cast Alloy Designation System

Alloy	Main Alloying Element
1xx.x	Pure aluminum, 99.00% max.
2xx.x	Copper
3xx.x	Silicon, with added copper and/or magnesium
4xx.x	Silicon
5xx.x	Magnesium
7xx.x	Zinc
8xx.x	Tin
9xx.x	Other elements
6xx.x	Unused series

A letter before the numerical designation indicates a modification of the original alloy or an impurity limit. These serial letters are assigned in alphabetical sequence starting with A, but omitting I, O, Q, and X, with X being reserved for experimental alloys. Note that explicit rules have been established for determining whether a proposed composition is a modification of an existing alloy or if it is a new alloy.

Experimental alloys of either the wrought or cast series are indicated by the addition of the prefix X. The prefix is dropped when the alloy is no longer experimental. However, during development and before an alloy is designated as experimental, a new composition may be identified by a serial number assigned by the originator. Use of the serial number is discontinued when the composition is registered with the Aluminum Association, Inc., and the ANSI H35.1 designation is assigned.

The compositions of a representative group of widely used commercial cast aluminum alloys are given in Table 4, taken from *Standards for Aluminum Sand and Permanent Mold Castings*[5] and other Aluminum Association and aluminum casting industry publications.

2.3 Aluminum Alloy Temper Designation System

The temper designation is always presented immediately following the alloy designation (see Sections 2.1 and 2.2), with a dash (-) between the two, e.g., 2014-T6.

The first character in the temper designation is a capital letter indicating the general class of treatment as follows:

F = As-Fabricated. Applies to wrought or cast products made by shaping processes in which there is no special control over thermal conditions or strain-hardening processes employed to achieve specific properties. For wrought alloys there are no mechanical property limits associated with this temper, though for cast alloys there may be.

O = Annealed. Applies to wrought products that are annealed to obtain the lower strength temper, usually to increase subsequent workability, and to cast products that are annealed to improve ductility and dimensional stability. The O may be followed by a digit other than zero.

H = Strain Hardened. Applies to products that have their strength increased by strain hardening. They may or may not have supplementary thermal treatments to produce some reduction in strength. The H is always followed by two or more digits.

W = Solution Heat treated. Applies only to alloys that age spontaneously after solution heat treating. This designation is specific only when digits are used in combination with W to indicate the period of natural aging, viz. W $\frac{1}{2}$ h.

T = Thermally Treated to Produce Stable Tempers other Than F. Applies to products that are thermally treated, with or without supplementary strain hardening, to produce stable tempers. The T is always followed by one or more digits.

The most widely used temper designations above are the H and T categories, and these are always followed by from one to four numeric digits that provide

Table 4 Nominal Compositions of Aluminum Alloy Castings[a]

	Percent of Alloying Elements[b]								
Alloy	Silicon	Copper	Manganese	Magnesium	Chromium	Nickel	Zinc	Titanium	Notes[c]
201.0	—	4.6	0.35	0.35	—	—	—	0.25	1
204.0	—	4.6	—	0.25	—	—	—	—	
A206.0	—	4.6	0.35	0.25	—	—	—	0.22	
208.0	3.0	4.0	—	—	—	—	—	—	
213.0	2.0	7.0	—	—	—	—	2.5	—	2
222.0	—	10.0	—	0.25	—	—	—	—	
224.0	—	5.0	0.35	—	—	—	—	—	3
240.0	—	8.0	0.5	6.0	—	0.5	—	—	
242.0	—	4.0	—	1.5	—	2.0	—	—	
A242.0	—	4.1	—	1.4	0.20	2.0	—	0.14	
295.0	1.1	4.5	—	—	—	—	—	—	
308.0	5.5	4.5	—	—	—	—	—	—	
319.0	6.0	3.5	—	—	—	—	—	—	
328.0	8.0	1.5	0.40	0.40	—	—	—	—	
332.0	9.5	3.0	—	1.0	—	—	—	—	
333.0	9.0	3.5	—	0.28	—	—	—	—	
336.0	12.0	1.0	—	1.0	—	2.5	—	—	
354.0	9.0	1.8	—	0.5	—	—	—	—	
355.0	5.0	1.25	—	0.5	—	—	—	—	
C355.0	5.0	1.25	—	0.5	—	—	—	—	4
356.0	7.0	—	—	0.32	—	—	—	—	
A356.0	7.0	—	—	0.35	—	—	—	—	4
357.0	7.0	—	—	0.52	—	—	—	—	
A357.0	7.0	—	—	0.55	—	—	—	0.12	4, 5
359.0	9.0	—	—	0.6	—	—	—	—	
360.0	9.5	—	—	0.5	—	—	—	—	
A360.0	9.5	—	—	0.5	—	—	—	—	4
380.0	8.5	3.5	—	—	—	—	—	—	
A380.0	8.5	3.5	—	—	—	—	—	—	4
383.0	10.5	2.5	—	—	—	—	—	—	
384.0	11.2	3.8	—	—	—	—	—	—	
B390.0	17.0	4.5	—	0.55	—	—	—	—	
413.0	12.0	—	—	—	—	—	—	—	
A413.0	12.0	—	—	—	—	—	—	—	
443.0	5.2	—	—	—	—	—	—	—	
B443.0	5.2	—	—	—	—	—	—	—	4
C443.0	5.2								6
A444.0	7.0	—	—	—	—	—	—	—	
512.0	1.8	—	—	4.0	—	—	—	—	
513.0	—	—	—	4.0	—	—	1.8	—	
514.0	—	—	—	4.0	—	—	—	—	
518.0	—	—	—	8.0	—	—	—	—	
520.0	—	—	—	10.0	—	—	—	—	
535.0	—	—	0.18	6.8	—	—	—	0.18	7
705.0	—	—	0.5	1.6	0.30	—	3.0	—	
707.0	—	—	0.50	2.1	0.30	—	4.2	—	
710.0	—	0.50	—	0.7	—	—	6.5	—	
711.0	—	0.50	—	0.35	—	—	6.5	—	8
712.0	—	—	—	0.58	0.50	—	6.0	0.20	
713.0	—	0.7	—	0.35	—	—	7.5	—	
771.0	—	—	—	0.9	0.40	—	7.0	0.15	
850.0	—	1.0	—	—	—	1.0	—	—	9
851.0	2.5	1.0	—	—	—	0.50	—	—	9
852.0	—	2.0	—	0.75	—	1.2	—	—	9

Table 4 (*Continued*)

[a] Based upon industry handbooks,$^{5-8}$ consult those references for specified limits. Values are nominal, i.e., average of range of limits for elements for which a range is shown; values are representative of separately cast test bars, not of specimens taken from commercial castings.

[b] Aluminum and normal impurities constitute remainder.

[c] 1. Also contains 0.7% silver.
2. Also contains 1.2% iron.
3. Also contains 0.10% vanadium and 0.18% zirconium.
4. Impurity limits are much lower for this alloy than for alloy listed above it.
5. Also contains 0.055% beryllium.
6. Also contains up to 2.0% total Iron.
7. Also contains 0.005% beryllium and 0.005% boron.
8. Also contains 1.0 iron.
9. Also contains 6.2% tin.

more detail about how the alloy has been fabricated. It is beyond the scope of this volume to describe the system in further detail and the reader if referred to refs. 1, 4, and 5 for additional needed information.

3 PROPERTIES OF ALUMINUM ALLOYS

The properties of a representative group of wrought aluminum alloys are shown in Tables 5 and 6. The properties of a representative group of cast aluminum alloys are shown in Tables 7 and 8.

4 APPLICATIONS OF ALUMINUM ALLOYS

There are at least two approaches to overviewing important applications of aluminum alloys: *by alloy class,* as initiated in Sections 1.1 and 1.2 and carried out in greater detail below, and *by type of application*. We will consider both approaches in the information below, reviewing by alloy class in Section 4.1 and by application in Section 4.2.

All photographs in this chapter are courtesy of the Aluminum Association, Inc. For a more complete discussion of these and other applications of aluminum alloys, the reader is referred to D. G. Altenpohl's very complete treatise on the aluminum industry, *Aluminum: Technology, Applications, and Environment.*[9]

4.1 Applications by Alloy Class

Wrought Alloys

1xxx: Pure Al. The major characteristics of the 1xxx series are:

- Strain hardenable
- Exceptionally high formability, corrosion resistance, and electrical conductivity
- Typical ultimate tensile strength range: 10–27 ksi
- Readily joined by welding, brazing, soldering

The 1xxx series represents the commercially pure aluminum, ranging from the baseline 1100 (99.00% min. Al) to relatively purer 1050/1350 (99.50% min.

Table 5 Typical Mechanical Properties of Wrought Aluminum Alloys[a]

	Tension							
Alloy & Temper	Ultimate Strength (ksi)	Yield Strength (ksi)	Elongation (%) in 2 in[b]	Elongation (%) in 4D[c]	Hardness Brinell Number (500 kg/10 mm)	Shear Ultimate Strength (ksi)	Fatigue Endurance Limit[d] (ksi)	Modulus[e] of Elasticity (10^3 ksi)
1060-O	10	4	43	—	19	7	3	10.0
1060-H12	12	11	16	—	23	8	4	10.0
1060-H14	14	13	12	—	26	9	5	10.0
1060-H16	16	15	8	—	30	10	6.5	10.0
1060-H18	19	18	6	—	35	11	6.5	10.0
1100-O	13	5	35	45	23	9	5	10.0
1100-H12	16	15	12	25	28	10	6	10.0
1100-H14	18	17	9	20	32	11	7	10.0
1100-H16	21	20	6	17	38	12	9	10.0
1100-H18	24	22	5	15	44	13	9	10.0
1350-O	12	4	23[f]	—	—	8	—	10.0
1350-H12	14	12	—	—	—	9	—	10.0
1350-H14	16	14	—	—	—	10	—	10.0
1350-H16	18	16	—	—	—	11	—	10.0
1350-H19	27	24	1.5[f]	—	—	15	—	10.0
2008-T4	36	18	28		—	21	—	10.2
2010-T4	35	19	25			21	8	10.2
2011-T3	55	43	—	15	96	32	18	10.2
2011-T8	59	45	—	12	100	35	18	10.2
2014-O	27	14	—	18	45	18	13	10.6
2014-T4, T451	62	42	—	20	105	38	20	10.6
2014-T6, T651	70	60	—	13	135	42	18	10.6
2017-O	26	10	—	22	45	18	13	10.5
2017-T4, T451	62	40	—	22	105	38	18	10.5
2024-O	27	11	20	22	47	18	13	10.6
2024-T3	70	50	18	—	120	41	20	10.6
2024-T4, T351	68	47	20	19	120	41	20	10.6
2024-T361	72	57	13	—	130	42	18	10.6
2025-T6	58	37	—	19	110	35	18	10.4
2036-T4	49	28	24	—	—	—	18[g]	10.3
2117-T4	43	24	—	27	70	28	14	10.3
2124-T851	70	64	—	8	—	—	—	10.6
2195-T351	52	36	17	—	—	—	—	10.6
2195-T851	66	51	10	—	—	—	—	10.6
2219-O	25	11	18	—	—	—	—	10.6
2219-T62	60	42	10	—	—	—	15	10.6
2219-T81, T851	66	51	10	—	—	—	15	10.6
2219-T87	69	57	10	—	—	—	15	10.6
2618-T61	64	54		10	115	38	18	10.8
3003-O	16	6	30	40	28	11	7	10.0
3003-H12	19	18	10	20	35	12	8	10.0
3003-H14	22	21	8	16	40	14	9	10.0
3003-H16	26	25	5	14	47	15	10	10.0
3003-H18	29	27	4	10	55	16	10	10.0
3004-O	26	10	20	25	45	16	14	10.0
3004-H32	31	25	10	17	52	17	15	10.0
3004-H34	35	29	9	12	63	18	15	10.0
3004-H36	38	33	5	9	70	20	16	10.0
3004-H38	41	36	5	6	77	21	16	10.0
3105-O	17	8	24	—	—	12	—	10.0
3105-H12	22	19	7	—	—	14	—	10.0
3105-H14	25	22	5	—	—	15	—	10.0
3105-H16	28	25	4	—	—	16	—	10.0
3105-H18	31	28	3	—	—	17	—	10.0
3105-H25	26	23	8	—	—	15	—	10.0
4032-T6	55	46		9	120	38	16	11.4
5005-O	18	6	25	—	28	11	—	10.0
5005-H32	20	17	11	—	36	14	—	10.0
5005-H34	23	20	8	—	41	14	—	10.0
5005-H36	26	24	6	—	46	15	—	10.0
5005-H38	29	27	6	—	51	16	—	10.0

Table 5 (*Continued*)

	Tension							
Alloy & Temper	Ultimate Strength (ksi)	Yield Strength (ksi)	Elongation (%) in 2 in[b]	Elongation (%) in 4D[c]	Hardness Brinell Number (500 kg/10 mm)	Shear Ultimate Strength (ksi)	Fatigue Endurance Limit[d] (ksi)	Modulus[e] of Elasticity (10^3 ksi)
5050-O	21	8	24	—	36	15	12	10.0
5050-H32	25	21	9	—	46	17	13	10.0
5050-H34	28	24	8	—	53	18	13	10.0
5050-H36	30	26	7	—	58	19	14	10.0
5050-H38	32	29	6	—	63	20	14	10.0
5052-O	28	13	25	30	47	18	16	10.2
5052-H32	33	28	12	18	60	20	17	10.2
5052-H34	38	31	10	14	68	21	18	10.2
5052-H36	40	35	8	10	73	23	19	10.2
5052-H38	42	37	7	8	77	24	20	10.2
5056-O	42	22	—	35	65	26	20	10.3
5056-H18	63	59	—	10	105	34	22	10.3
5056-H38	60	50	—	15	100	32	22	10.3
5083-O	42	21	—	22	—	25	—	10.3
5083-H116	46	33	—	16	—	—	23	10.3
5083-H321	46	33	—	16	—	—	23	10.3
5086-O	38	17	22	—	—	23	—	10.3
5086-H32	42	30	12	—	—	—	—	10.3
5086-H34	47	37	10	—	—	27	—	10.3
5086-H116	42	30	12	—	—	—	—	10.3
5154-O	35	17	27	—	58	22	17	10.2
5154-H32	39	30	15	—	67	22	18	10.2
5154-H34	42	33	13	—	73	24	19	10.2
5154-H36	45	36	12	—	78	26	20	10.2
5154-H38	48	39	10	—	80	28	21	10.2
5454-O	36	17	22	—	62	23	—	10.2
454-H32	40	30	10	—	73	24	—	10.2
5454-H34	44	35	10	—	81	26	—	10.2
5454-H111	38	26	14	—	70	23	—	10.2
5456-O	45	23	—	24		—	—	10.3
5456-H116	51	37	—	16	90	30	—	10.3
5456-H321	51	37	—	16	90	30	—	10.3
5657-H25	23	20	12	—	40	12	—	10.0
5657-H28, H38	28	24	7	—	50	15	—	10.0
5754-O	32	14	26	—	—	19	—	10.3
6009-T4	32	18	25	—	—	19	—	10.0
6022-T4	37	22	26	—	—	22	—	10.0
6061-O	18	8	25	30	30	12	9	10.0
6061-T4, T451	35	21	22	25	65	24	14	10.0
6061-T6, T651	45	40	12	17	95	30	14	10.0
6063-O	13	7	—	—	25	10	8	10.0
6063-T4	25	13	22	—	—	—	—	10.0
6063-T5	27	21	12	—	60	17	10	10.0
6063-T6	35	31	12	—	73	22	10	10.0
6063-T83	37	35	9	—	82	22	—	10.0
6066-O	22	12	—	18	43	14	—	10.0
6066-T4, T451	52	30	—	18	90	29	—	10.0
6066-T6, T651	57	52	—	12	120	34	16	10.0
6070-T6	55	51	10	—	—	34	14	10.0
6101-H111	14	11	—	—	—	—	—	10.0
6101-T6	32	28	15	—	71	20	—	10.0
6111-T4	42	22	26	—	—	25	—	10.0
6111-T41	39	22	26	—	—	23	—	10.0
6262-T9	58	55		10	120	35	13	10.0
6351-T4	36	22	20	—	—	44	—	10.0
6351-T6	45	41	14	—	95	29	13	10.0
7049-T73	75	65	—	12	135	44	—	10.4
7049-T7352	75	63	—	11	135	43		10.4

Table 5 (*Continued*)

Alloy & Temper	Tension				Hardness Brinell Number (500 kg/10 mm)	Shear Ultimate Strength (ksi)	Fatigue Endurance Limit[d] (ksi)	Modulus[e] of Elasticity (10^3 ksi)
	Ultimate Strength (ksi)	Yield Strength (ksi)	Elongation (%) in 2 in[b]	Elongation (%) in 4D[c]				
7050-T7351X	72	63	—	12	—	—	—	10.4
7050-T7451	76	68	—	11	—	44	—	10.4
7050-T7651	80	71	—	11	—	47	—	10.4
7075-O	33	15	17	16	60	22		10.4
7075-T6, T651	83	73	11	11	150	48	23	10.4
7175-T74	76	66		11	135	42	23	10.4
7178-O	33	15	16	—	—	—	—	10.4
7178-T6, T651	88	78	11	—	—	—	—	10.4
7178-T76, T7651	83	73	11	—	—	—	—	10.4
7475-T61	82	71	11	—	—	—	—	10.4
7475-T651	85	74	—	13	—	—	—	10.4
7475-T7351	72	61	—	13	—	—	—	10.4
7475-T761	75	65	12	—	—	—	—	10.4
7475-T7651	77	67	—	12	—	—	—	10.4
8176-H24	17	14	15	—	80	—	—	10.0

[a] Based upon industry handbooks.[1,16,17] Consult those references for limits. For tensile yield strengths, offset = 0.2%.

[b] Elongation measured over a 2 in. gauge length on $\frac{1}{16}$-in thick sheet-type specimens.

[c] Elongation measured over 2 in. gauge length (4D) in $\frac{1}{2}$-in. diameter specimens.

[d] Based on 500,000,000 cycles of completely reversed stress using R. R. Moore type of machines and specimens.

[e] Average of tension and compression moduli; compressive modulus is nominally about 2% greater than the tension modulus.

[f] Measured over a 10-in. gauge length in wire.

[g] At 10^7 cycles with flexural fatigue specimens.

Table 6 Typical Mechanical Properties of Wrought Aluminum Alloys—Metric[a]

Alloy & Temper	Tension				Hardness Brinell Number (500 kg/10 mm)	Shear Ultimate Strength (MPa)	Fatigue Endurance Limit[d] (MPa)	Modulus[e] of Elasticity (GPa)
	Ultimate Strength (MPa)	Yield Strength (MPa)	Elongation (%) in 50 mm[b]	Elongation (%) in 5D[c]				
1060-O	70	30	43	—	19	50	20	69
1060-H12	85	75	16	—	23	55	30	69
1060-H14	100	90	12	—	26	60	35	69
1060-H16	115	105	8	—	30	70	45	69
1060-H18	130	125	6	—	35	75	45	69
1100-O	90	35	35	42	23	60	35	69
1100-H12	110	105	12	22	28	70	40	69
1100-H14	125	115	9	18	32	75	50	69
1100-H16	145	140	6	15	38	85	60	69
1100-H18	165	150	5	13	44	90	60	69
1350-O	85	30	23[f]	—	—	55	—	69
1350-H12	95	85	—	—	—	60	—	69
1350-H14	110	95	—	—	—	70	—	69
1350-H16	125	110	—	—	—	75	—	69
1350-H19	185	165	1.5[f]	—	—	105	—	69
2008-T4	250	125	28	—	145	70	—	70
2010-T4, T41	240	130	25	—	145	70	—	70
2011-T3	380	295	—	13	95	220	125	70
2011-T8	405	310	—	10	100	240	125	70
2014-O	185	95	—	16	45	125	90	73
2014-T4, T451	425	290	—	18	105	260	140	73
2014-T6, T651	485	415	—	11	135	290	125	73

Table 6 (*Continued*)

Alloy & Temper	Tension				Hardness Brinell Number (500 kg/10 mm)	Shear Ultimate Strength (MPa)	Fatigue Endurance Limit[d] (MPa)	Modulus[e] of Elasticity (GPa)
	Ultimate Strength (MPa)	Yield Strength (MPa)	Elongation (%)					
			in 50 mm[b]	in 5D[c]				
2017-O	180	70	—	20	45	125	90	73
2017-T4, T451	425	275	—	20	105	260	125	73
2024-O	185	75	20	20	47	125	90	73
2024-T3	485	345	18	—	120	285	140	73
2024-T4, T351	472	325	20	17	120	285	140	73
2024-T361	495	395	13	—	130	290	125	73
2025-T6	400	255	—	17	110	240	125	72
2036-T4	340	195	24	—	—	205	125[g]	71
2117-T4	295	165	—	24	70	195	95	71
2124-T851	485	440	—	8	—	—	—	73
2195-T351	360	250	17	—	—	—	—	73
2195-T851	455	350	10	—	—	—	—	73
2219-O	170	75	18	—	—	—	—	73
2219-T62	415	290	10	—	—	—	105	73
2219-T81, T851	455	350	10	—	—	—	105	73
2219-T87	475	395	10	—	—	—	105	73
2618-T61	440	370		10	115	260	90	73
3003-O	110	40	30	37	28	75	50	69
3003-H12	130	125	10	18	35	85	55	69
3003-H14	150	145	8	14	40	95	60	69
3003-H16	175	170	5	12	47	105	70	69
3003-H18	200	185	4	9	55	110	70	69
3004-O	180	70	20	22	45	110	95	69
3004-H32	215	170	10	15	52	115	105	69
3004-H34	240	200	9	10	63	125	105	69
3004-H36	260	230	5	8	70	140	110	69
3004-H38	285	250	5	5	77	145	110	69
3105-O	115	55	24	—	—	85	—	69
3105-H12	150	130	7	—	—	95	—	69
3105-H14	170	150	5	—	—	105	—	69
3105-H16	195	170	4	—	—	110	—	69
3105-H18	215	195	3	—	—	115	—	69
3105-H25	180	160	8	—	—	95	—	69
4032-T6	380	315		9	120	260	110	79
5005-O	125	40	25	—	28	75	—	69
5005-H32	140	115	11	—	36	95	—	69
5005-H34	160	140	8	—	41	95	—	69
5005-H36	180	165	6	—	46	105	—	69
5005-H38	200	185	5	—	51	110	—	69
5050-O	145	55	24	—	36	105	85	69
5050-H32	170	145	9	—	46	115	90	69
5050-H34	190	165	8	—	53	125	90	69
5050-H36	205	180	7	—	58	130	95	69
5050-H38	220	200	6	—	63	140	95	69
5052-O	195	90	25	27	47	125	110	70
5052-H32	230	195	12	16	60	140	115	70
5052-H34	260	215	10	12	68	145	125	70
5052-H36	275	240	8	9	73	160	130	70
5052-H38	290	255	7	7	77	165	140	70
5056-O	290	150	—	32	65	180	140	71
5056-H18	435	405	—	9	105	235	150	71
5056-H38	415	345	—	13	100	220	150	71
5083-O	290	145	—	20	—	170	—	71
5083-H116	315	230	—	14	—	—	160	71
5083-H321	315	230	—	14	—	—	160	71
5086-O	260	115	22	—	—	165	—	71
5086-H32	290	205	12	—	—	—	—	71
5086-H34	325	255	10	—	—	185	—	71
5086-H116	290	205	12	—	—	—	—	71

Table 6 (*Continued*)

	Tension							
			Elongation (%)					
Alloy & Temper	Ultimate Strength (MPa)	Yield Strength (MPa)	in 50 mm[b]	in 5D[c]	Hardness Brinell Number (500 kg/10 mm)	Shear Ultimate Strength (MPa)	Fatigue Endurance Limit[d] (MPa)	Modulus[e] of Elasticity (GPa)
5154-O	240	115	27	—	58	150	115	70
5154-H32	270	205	15	—	67	150	125	70
5154-H34	290	230	13	—	73	165	130	70
5154-H36	310	250	12	—	78	180	140	70
5154-H38	330	270	10	—	80	195	145	70
5454-O	250	115	22	—	62	160	—	70
5454-H32	275	205	10	—	73	165	—	70
5454-H34	305	240	10	—	81	180	—	70
5454-H111	260	180	14	—	70	160	—	70
5456-O	310	160	—	22		—	—	71
5456-H116	350	255	—	14	90	205	—	71
5456-H321	350	255	—	14	90	205	—	71
5657-H25	160	140	12	—	40	95	—	69
5657-H28, H38	195	165	7	—	50	105	—	69
5754-O	220	100	26	—	—	130	—	71
6009-T4	220	125	25	—	—	130	—	69
6061-O	125	55	25	27	30	85	60	69
6061-T4, T451	240	145	22	22	65	165	95	69
6061-T6, T651	310	275	12	15	95	205	95	69
6063-O	90	50	—	—	25	70	55	69
6063-T4	170	90	22	—	—	—	—	69
6063-T5	185	145	12	—	60	115	70	69
6063-T6	240	215	12	—	73	150	70	69
6063-T83	255	240	9	—	82	150	—	69
6066-O	150	85	—	16	43	95	—	69
6066-T4, T451	360	205	—	16	90	200	—	69
6066-T6, T651	395	360	—	10	120	235	110	69
6070-T6	380	350	10	—	—	235	95	69
6101-H111	95	75	—	—	—	—	—	69
6101-T6	220	195	15(&)	—	71	140	—	69
6111-T4	280	150	26	—	—	175	—	69
6111-T41	270	150	26	—	—	160	—	69
6262-T9	400	380	—	9	120	240	90	69
6351-T4	250	150	20	—	—	44	—	69
6351-T6	310	285	14	—	95	200	90	69
7049-T73	515	450	—	10	135	305	—	72
7049-T7352	515	435	—	9	135	295	—	72
7050-T7351X	495	435	—	11	—	—	—	72
7050-T7451	525	470	—	10	—	305	—	72
7050-T7651	550	490	—	10	—	325	—	72
7075-O	230	105	17	14	60	150		72
7075-T6, T651	570	505	11	9	150	330	160	72
7175-T74	525	455	—	10	135	290	160	72
7178-O	230	105	15	14	—	—	—	72
7178-T6, T651	605	540	10	9	—	—	—	72
7178-T76, T7651	570	505	11	9	—	—	—	71
7475-T61	565	490	11	—	—	—	—	70
7475-T651	585	510	—	13	—	—	—	72
7475-T7351	495	420	—	13	—	—	—	72
7475-T761	515	450	12	—	—	—	—	70
7475-T7651	530	460	—	12	—	—	—	72
8176-H24	160	95	15	—	—	70	—	10

Table 6 (*Continued*)

[a] Based upon industry handbooks.[1,16,17] Consult those references for limits. For tensile yield strengths, offset = 0.2%.

[b] Elongation measured over a 500-mm. gauge length on 1.60-mm thick sheet-type specimens.

[c] Elongation measured over 500-mm gauge length (5D) in 12.5-mm diameter specimens.

[d] Based on 500,000,000 cycles of completely reversed stress using R.R. Moore type of machines and specimens.

[e] Average of tension and compression moduli; compressive modulus is nominally about 2% greater.

[f] Measured over a 250-mm gauge length in wire.

[g] At 10^7 cycles with flexural fatigue specimens.

Table 7 Typical Mechanical Properties of Wrought Aluminum Alloy Castings[a]

	Tension						
Alloy & Temper	Ultimate Strength (ksi)	Yield Strength (ksi)	Elongation in 2 in or 4D (%)	Hardness Brinell Number (500 kg / 10 mm)	Shear Ultimate Strength (ksi)	Fatigue Endurance Limit[b] (ksi)	Modulus[c] of Elasticity (10^3 ksi)
			Sand Cast				
201.0-T6	65	55	8	130	—	—	—
201-0-T7	68	60	6	—	—	14	—
201.0-T43	60	37	17	—	—	—	—
204.0-T4	45	28	6	—	—	—	—
A206.0-T4	51	36	7	—	40	—	—
208.0-F	21	14	3	—	17	11	—
213.0-F	24	15	2	70	20	9	—
222.0-O	27	20	1	80	21	9.5	
222.0-T61	41	40	<0.5	115	32	8.5	10.7
224.0-T72	55	40	10	123	35	9	10.5
240.0-F	34	28	1	90	—	—	—
242.0-F	31	20	1	—	—	—	10.3
242.0-O	27	18	1	70	21	8	10.3
242.0-T571	32	30	1	85	26	11	10.3
242.0-T61	32	20	—	90–120	—	—	10.3
242.0-T77	30	23	2	75	24	10.5	10.3
A242.0-T75	31	—	2	—	—	—	—
295.0-T4	32	16	9	80	26	7	10.0
295.0-T6	36	24	5	75	30	7.5	10.0
295.0-T62	41	32	2	90	33	8	10.0
295.0-T7	29	16	3	55–85	—	—	10.0
319-F	27	18	2	70	22	10	10.7
319.0-T5	30	26	2	80	24	11	10.7
319.0-T6	36	24	2	80	29	11	10.7
328.0-F	25	14	1	45–75	—	—	—
328.0-T6	34	21	1	65–95	—	—	—
355.0-F	23	12	3	—	—	—	10.2
355.0-T51	28	23	2	65	22	8	10.2
355.0-T6	35	25	3	80	28	9	10.2
355.0-T61	35	35	1	90	31	9.5	10.2
355.0-T7	38	26	1	85	28	10	10.2
355.0-T71	35	29	2	75	26	10	10.2
C355.0-T6	39	29	5	85	—	—	—
356.0-F	24	18	6	—	—	—	10.5
356.0-T51	25	20	2	60	20	8	10.5
356.0-T6	33	24	4	70	26	8.5	10.5
356.0-T7	34	30	2	75	24	9	10.5
356.0-T71	28	21	4	60	20	8.5	10.5
A356.0-F	23	12	6	—	—	—	10.5
A356.0-T51	26	18	3	—	—	—	10.5
A356.0-T6	40	30	6	75	—	—	10.5
A356.0-T71	30	20	3	—	—	—	10.5

Table 7 (*Continued*)

	Tension						
Alloy & Temper	Ultimate Strength (ksi)	Yield Strength (ksi)	Elongation in 2 in or 4D (%)	Hardness Brinell Number (500 kg/10 mm)	Shear Ultimate Strength (ksi)	Fatigue Endurance Limit[b] (ksi)	Modulus[c] of Elasticity (10^3 ksi)
357.0-F	25	13	5	—	—	—	—
357.0-T51	26	17	3	—	—	—	—
357.0-T6	50	43	2	—	—	—	—
357.0-T7	40	34	3	60	—	—	—
A357.0-T6	46	36	3	85	40	12	—
359.0-T62	50	42	6	—	—	—	—
A390.0-F	26	26	<1.0	100	—	—	—
A390.0-T5	26	26	<1.0	100	—	—	—
A390.0-T6	40	40	<1.0	140	—	13	—
A390.0-T7	36	36	<1.0	115	—	—	—
443.0-F	19	8	8	40	14	8	10.3
B443.0-F	17	6	3	25–55	—	—	—
A444.0-F	21	9	9	44	—	—	—
A444.0-T4	23	9	12	45			
511.0-F	21	12	3	50	17	8	—
512.0-F	20	13	2	50	17	9	—
514.0-F	25	12	9	50	20	7	—
520.0-T4	48	26	16	75	34	8	—
535.0-F	35	18	9	60–90	—	—	—
535.0-T5	35	18	9	60–90	—	—	—
A535.0-F	36	18	9	65	—	—	—
707.0-T5	33	22	2	70–100	—	—	—
707.0-T7	37	30	1	65–95	—	—	—
710.0-F	32	20	2	60–90	—	—	—
710.0-T5	32	20	2	60–90	—	—	—
712.0-F	34	25	4	60–90	—	—	—
712.0-T5	34	25	4	60–90	—	—	—
713.0-F	32	22	3	60–90	—	—	—
713.0-T5	32	22	3	60–90			
771.0-T5	32	27	3	70–100	—	—	—
771.0-T52	36	30	2	70–100	—	—	—
771.0-T53	36	27	2	—	—	—	—
771.0-T6	42	35	5	75–105	—	—	—
771.0-T71	48	45	2	105–135	—	—	—
850.0-T5	20	11	8	45	14	—	10.3
851.0-T5	20	11	5	45	14	—	10.3
852.0-T5	27	22	2	65	18	10	10.3
Permanent Mold							
201.0-T6	65	55	8	130	—	—	—
201-0-T7	68	60	6	—	—	14	—
201.0-T43	60	37	17	—	—	—	—
204.0-T4	48	29	8	—	—	—	—
A206.0-T4	62	38	17	—	42	—	—
A206.0-T7	63	50	12	—	37	—	—
208.0-T6	35	22	2	75–105	—	—	—
208.0-T7	33	16	3	65–95	—	—	—
213.0-F	30	24	2	85	24	9.5	—
222.0-T551	37	35	<0.5	115	30	8.5	10.7
222.0-T52	35	31	1	100	25	—	10.7
238.0-F	30	24	2	100	24	—	—
242.0-T571	40	34	1	105	30	10.5	10.3
242.0-T61	47	42	1	110	35	10	10.3
A249.0-T63	69	60	6	—	—	—	—
296.0-T7	39	20	5	80	30	9	10.1
308.0-F	28	16	2	70	22	13	—

Table 7 (*Continued*)

	Tension			Hardness Brinell Number (500 kg/10 mm)	Shear Ultimate Strength (ksi)	Fatigue Endurance Limit^b (ksi)	Modulus^c of Elasticity (10^3 ksi)
Alloy & Temper	Ultimate Strength (ksi)	Yield Strength (ksi)	Elongation in 2 in or 4D (%)				
319.0-F	34	19	3	85	24	—	10.7
319.0-T6	40	27	3	95	—	—	10.7
324.0-F	30	16	4	70	—	—	—
324.0-T5	36	26	3	90	—	—	—
324.0-T62	45	39	3	105	—	—	—
332.0-T5	36	28	1	105	—	—	—
328.0-T6	34	21	1	65–95	—	—	—
333.0-F	34	19	2	90	27	15	—
333.0-T5	34	25	1	100	27	12	—
333.0-T6	42	30	2	105	33	15	—
333.0-T7	37	28	2	90	28	12	—
336.0-T551	36	28	1	105	28	14	—
336.0-T65	47	43	1	125	36	—	—
354.0-T61	48	37	3	—	—	—	—
354.0-T62	52	42	2	—	—	—	—
355.0-F	27	15	4	—	—	—	10.2
355.0-T51	30	24	2	75	24	—	10.2
355.0-T6	42	27	4	90	34	10	10.2
355.0-T61	45	40	2	105	36	10	10.2
355.0-T7	40	30	2	85	30	10	10.2
355.0-T71	36	31	3	85	27	10	10.2
C355.0-T6	48	28	8	90	—	—	10.2
C355.0-T61	46	34	6	100	—	—	10.2
C355.0-T62	48	37	5	100	—	—	10.2
356.0-F	26	18	5	—	—	—	10.5
356.0-T51	27	20	2	—	—	—	10.5
356.0-T6	38	27	5	80	30	13	10.5
356.0-T7	32	24	6	70	25	11	10.5
356.0-T71	25	—	3	60–90	—	—	10.5
A356.0-F	27	13	8	—	—	—	10.5
A356.0-T51	29	20	5	—	—	—	10.5
A356.0-T6	41	30	12	80	—	—	10.5
357.0-F	28	15	6	—	—	—	—
357.0-T51	29	21	4	—	—	—	—
357.0-T6	52	43	5	100	35	13	—
357.0-T7	38	30	5	70	—	—	—
A357.0-T6	52	42	5	100	35	15	—
359.0-T61	48	37	6	—	—	—	—
359.0-T62	50	42	6	—	—	16	—
A390.0-F	29	29	<1.0	110	—	—	—
A390.0-T5	29	29	<1.0	110	—	—	—
A390.0-T6	45	45	<1.0	145	—	17	—
A390.0-T7	38	38	<1.0	120	—	15	—
443.0-F	23	9	10	45	16	8	10.3
B443.0-F	21	6	6	30–60	—	—	—
A444.0-F	24	11	13	44	—	—	—
A444.0-T4	23	10	21	45	16	8	—
513.0-F	27	16	7	60	22	10	—
535.0-F	35	18	8	60–90	—	—	—
705.0-T5	37	17	10	55–75	—	—	—
707.0-T7	45	35	3	80–110	—	—	—
711.0-T1	28	18	7	55–85	—	—	—
713.0-T5	32	22	4	60–90			
850.0-T5	23	11	12	45	15	9	10.3
851.0-T5	20	11	5	45	14	9	10.3
851.0-T6	18	—	8	—	—	—	10.3
852.0-T5	32	23	5	70	21	11	10.3

Table 7 (*Continued*)

Alloy & Temper	Tension			Hardness Brinell Number (500 kg/10 mm)	Shear Ultimate Strength (ksi)	Fatigue Endurance Limit[b] (ksi)	Modulus[c] of Elasticity (10^3 ksi)
	Ultimate Strength (ksi)	Yield Strength (ksi)	Elongation in 2 in or 4D (%)				
Die Cast							
360.0-F	44	25	3	75	28	20	10.3
A360.0-F	46	24	4	75	26	18	10.3
380.0-F	46	23	3	80	28	20	10.3
A380.0-F	47	23	4	80	27	20	10.3
383.0-F	45	22	4	75	—	21	10.3
384.0-F	48	24	3	85	29	20	—
390.0-F	40.5	35	<1	—	—	—	—
B390.0-F	46	36	<1	120	—	20	11.8
392.0-F	42	39	<1	—	—	—	—
413.0-F	43	21	3	80	25	19	10.3
A413.0-F	42	19	4	80	25	19	—
C443.0-F	33	14	9	65	29	17	10.3
518.0-F	45	28	5	80	29	20	—

[a] Based upon industry handbooks.[5–8] Consult those references for limits. Values are representative of separately cast test bars, not of specimens taken from commercial castings. For tensile yield strengths, offset = 0.2%.

[b] Based on 500,000,000 cycles of completely reversed stress using R. R. Moore type of machines and specimens.

[c] Average of tension and compression moduli; compressive modulus is nominally about 2% greater than the tension modulus.

Table 8 Typical Mechanical Properties of Aluminum Alloy Castings—Metric[a]

Alloy & Temper	Tension			Hardness Brinell Number (500 kg/10 mm)	Shear Ultimate Strength (MPa)	Fatigue Endurance Limit[b] (MPa)	Modulus[c] of Elasticity (GPa)
	Ultimate Strength (MPa)	Yield Strength (MPa)	Elongation in 5D (%)				
Sand Cast							
201.0-T6	450	380	8	130	—	—	—
201-0-T7	470	415	6	—	—	95	—
201.0-T43	415	255	17	—	—	—	—
204.0-T4	310	195	6	—	—	—	—
A206.0-T4	350	250	7	—	275	—	—
2008-0-F	145	655	3	—	115	75	—
213.0-F	165	105	2	70	140	60	—
222-0-O	185	140	1	80	145	65	
222.0-T61	285	275	<0.5	115	220	60	74
224.0-T72	380	275	10	123	240	60	73
240.0-F	235	195	1	90	—	—	—
242.0-F	145	140	1	—	—	—	71
242.0-O	185	125	1	70	145	55	71
242.0-T571	220	205	1	85	180	75	71
242.0-T61	220	140	—	90–120	—	—	71
242.0-T77	205	160	2	75	165	70	71
A242.0-T75	215	—	2	—	—	—	—
295.0-T4	220	110	9	80	180	50	69
295.0-T6	250	165	5	75	205	50	69
295.0-T62	285	220	2	90	230	55	69
295.0-T7	200	110	3	55–85	—	—	69
319-F	185	125	2	70	150	70	74
319.0-T5	205	180	2	80	165	75	74
319.0-T6	250	165	2	80	200	75	74

Table 8 (*Continued*)

	Tension						
Alloy & Temper	Ultimate Strength (MPa)	Yield Strength (MPa)	Elongation in 5D (%)	Hardness Brinell Number (500 kg/10 mm)	Shear Ultimate Strength (MPa)	Fatigue Endurance Limit[b] (MPa)	Modulus[c] of Elasticity (GPa)
328.0-F	170	95	1	45–75	—	—	—
328.0-T6	235	145	1	65–95	—	—	—
355.0-F	160	85	3	—	—	—	70
355.0-T51	195	160	2	65	150	55	70
355.0-T6	240	170	3	80	195	60	70
355.0-T61	240	240	1	90	215	65	70
355.0-T7	260	180	1	85	195	70	70
355.0-T71	240	200	2	75	180	70	70
C355.0-T6	270	200	5	85	—	—	—
356.0-F	165	125	6	—	—	—	73
356.0-T51	170	140	2	60	140	55	73
356.0-T6	230	165	4	70	180	60	73
356.0-T7	235	205	2	75	165	60	73
356.0-T71	195	145	4	60	140	60	73
A356-0-F	160	85	6	—	—	—	73
A356.0-T51	180	125	3	—	—	—	73
A356.0-T6	275	205	6	75	—	—	73
A356.0-T71	205	140	3	—	—	—	73
357.0-F	170	90	5	—	—	—	—
357.0-T51	180	115	3	—	—	—	—
357.0-T6	345	295	2	—	—	—	—
357.0-T7	275	235	3	60	—	—	—
A357.0-T6	315	250	3	85	275	95	—
359.0-T62	345	290	6	16	—	—	—
A390.0-F	180	180	<1.0	100	—	—	—
A390.0-T5	180	180	<1.0	100	—	—	—
A390.0-T6	275	275	<1.0	140	—	90	—
A390.0-T7	250	250	<1.0	115	—	—	—
443.0-F	130	55	8	40	95	55	71
B443.0-F	115	40	3	25–55	—	—	—
A444.0-F	145	60	9	43,400	—	—	—
A444.0-T4	23	60	12				
511.0-F	145	85	3	50	115	55	
512.0-F	140	90	2	50	115	60	
514.0-F	170	85	9	50	140	50	—
520.0-T4	330	180	16	75	235	55	—
535.0-F	240	125	9	60–90	—	—	—
535-0-T5	240	125	9	60–90	—	—	—
A535.0-F	250	125	9	65	—	—	—
707.0-T5	230	150	2	70–100	—	—	—
707.0-T7	255	205	1	65–95	—	—	—
710.0-F	220	140	2	60–90	—	—	—
710.0-T5	220	140	2	60–90	—	—	—
712.0-F	235	170	4	60–90	—	—	—
712.0-T5	235	170	4	60–90	—	—	—
713.0-F	220	150	3	60–90	—	—	—
713.0-T5	220	150	3	60–90			
771.0-T5	220	185	3	70–100	—	—	—
771.0-T52	250	205	2	70–100	—	—	—
771.0-T53	250	185	2	—	—	—	—
771.0-T6	290	240	5	75–105	—	—	—
771.0-T71	330	310	2	105–135	—	—	—
850.0-T5	140	75	8	45	95	—	71
851.0-T5	140	75	5	45	95	—	71
852.0-T5	185	150	2	n 65	125	60	71

Table 8 (*Continued*)

	Tension						
Alloy & Temper	Ultimate Strength (MPa)	Yield Strength (MPa)	Elongation in 5D (%)	Hardness Brinell Number (500 kg/10 mm)	Shear Ultimate Strength (MPa)	Fatigue Endurance Limit[b] (MPa)	Modulus[c] of Elasticity (GPa)
				Permanent Mold			
201.0-T6	450	380	8	130	—	—	—
201-0-T7	470	415	6	—	—	95	—
201.0-T43	415	255	17	—	—	—	—
204.0-T4	330	200	8	—	—	—	—
A206.0-T4	430	260	17	—	290	—	—
A206.0-T7	435	345	12	—	255	—	—
208.0-T6	240	150	2	75–105	—	—	—
208.0-T7	230	110	3	65–95	—	—	—
213.0-F	205	165	2	85	165	65	—
222.0-T551	255	240	<0.5	115	205	60	74
222.0-T52	240	215	1	100	170	—	74
238.0-F	205	165	2	100	165	—	—
242.0-T571	275	235	1	105	205	70	74
242.0-T61	325	290	1	110	450	70	74
A249.0-T63	475	415	6	—	—	—	—
296.0-T7	270	140	5	80	205	60	70
308.0-F	195	110	2	70	150	90	—
319.0-F	235	130	3	85	165	—	74
319.0-T6	275	185	3	95	—	—	74
324.0-F	205	110	4	70	—	—	—
324.0-T5	250	180	3	90	—	—	—
324.0-T62	310	270	3	105	—	—	—
332.0-T5	250	195	1	105	—	—	—
328.0-T6	235	145	1	65–95	—	—	—
333.0-F	235	130	2	90	185	105	—
333.0-T5	235	170	1	100	185	85	—
333.0-T6	290	205	2	105	230	105	—
333.0-T7	255	195	2	90	195	85	—
336.0-T551	250	193	1	105	193	95	—
336.0-T65	325	295	1	125	250	—	—
354.0-T61	330	255	3	—	—	—	—
354.0-T62	360	290	2	—	—	—	—
355.0-F	185	105	4	—	—	—	70
355.0-T51	205	165	2	75	165	—	70
355.0-T6	290	185	4	90	235	70	70
355.0-T61	310	275	2	105	250	70	70
355.0-T7	275	205	2	85	205	70	70
355.0-T71	250	215	3	85	185	70	70
C355.0-T6	330	195	8	90	—	—	70
C355.0-T61	315	235	6	100	—	—	70
C355.0-T62	330	255	5	100	—	—	70
356.0-F	180	125	5	—	—	—	73
356.0-T51	185	140	2	—	—	—	73
356.0-T6	260	185	5	80	205	90	73
356.0-T7	220	165	6	70	170	75	73
356.0-T71	170	—	3	60–90	—	—	73
A356.0-F	185	90	8	—	—	—	73
A356.0-T51	200	140	5	—	—	—	73
A356.0-T6	285	205	12	80	—	—	73
357.0-F	195	105	6	—	—	—	—
357.0-T51	200	145	4	—	—	—	—
357.0-T6	360	295	5	100	240	90	—
357.0-T7	260	205	5	70	—	—	—
A357.0-T6	360	290	5	100	240	105	—
359.0-T61	330	255	6	—	—	—	—
359.0-T62	345	290	6	—	—	110	—
A390.0-F	200	200	<1.0	110	—	—	—
A390.0-75	200	200	<1.0	110	—	—	—

Table 8 (*Continued*)

Alloy & Temper	Tension			Hardness Brinell Number (500 kg/10 mm)	Shear Ultimate Strength (MPa)	Fatigue Endurance Limit[b] (MPa)	Modulus[c] of Elasticity (GPa)
	Ultimate Strength (MPa)	Yield Strength (MPa)	Elongation in 5D (%)				
A390.0-T6	310	310	<1.0	145	—	115	—
A390.0-T7	260	260	<1.0	120	—	105	—
443.0-F	160	60	10	45	110	55	71
B443.0-F	145	40	6	30–60	—	—	—
A444.0-F	165	75	13	44	—	—	—
A444.0-T4	160	70	21	45	110	55	—
513.0-F	185	110	7	60	150	70	—
535.0-F	240	125	8	60–90	—	—	—
705.0-T5	255	115	10	55–75	—	—	—
707.0-T7	310	240	3	80–110	—	—	—
711.0-T1	195	125	7	55–85	—	—	—
713.0-T5	220	150	4	60–90			
850.0-T5	160	75	12	45	105	60	71
851.0-T5	140	75	5	45	95	60	71
851.0-T6	125	—	8	—	—	—	71
852.0-T5	220	160	5	70	145	75	71
Die Cast							
A360.0-F	315	165	4	75	180	124	71
380.0-F	315	160	3	80	195	140	71
A380.0-F	325	160	4	80	185	140	71
383.0-F	310	150	4	75	—	145	71
384.0-F	330	165	3	85	200	140	—
390.0-F	280	240	<1	—	—	—	—
B390.0-F	315	250	<1	120	—	140	81
392.0-F	290	270	<1	—	—	—	—
413.0-F	295	145	3	80	170	130	71
A413.0-F	290	130	4	80	170	130	—
C443.0-F	230	95	9	65	200	115	71
518.0-F	310	193	5	80	200	140	—

[a] Based upon industry handbooks.[5–8] Consult those references for limits. Values are representative of separately cast test bars, not of specimens taken from commercial castings. For tensile yield strengths, offset = 0.2%.

[b] Based on 500,000,000 cycles of completely reversed stress using R. R. Moore type of machines and specimens.

[c] Average of tension and compression moduli; compressive modulus is nominally about 2% greater.

Al) and 1175 (99.75 % min. Al). The 1xxx series are strain hardenable but would not be used where strength is a prime consideration.

The primary uses of the 1xxx series would be applications where the combination of extremely high corrosion resistance and formability are required, e.g., foil and strip for packaging (Fig. 1), chemical equipment, tank car or truck bodies, spun hollowware, and elaborate sheet metal work.

Electrical applications are one major use of the 1xxx series, primarily 1350, which has relatively tight controls on those impurities that might lower electrical conductivity. As a result, an electrical conductivity of 62% of the International Annealed Copper Standard (IACS) is guaranteed for this material, which, combined with the natural light weight of aluminum, means a significant weight and therefore cost advantage over copper in electrical applications (Fig. 2).

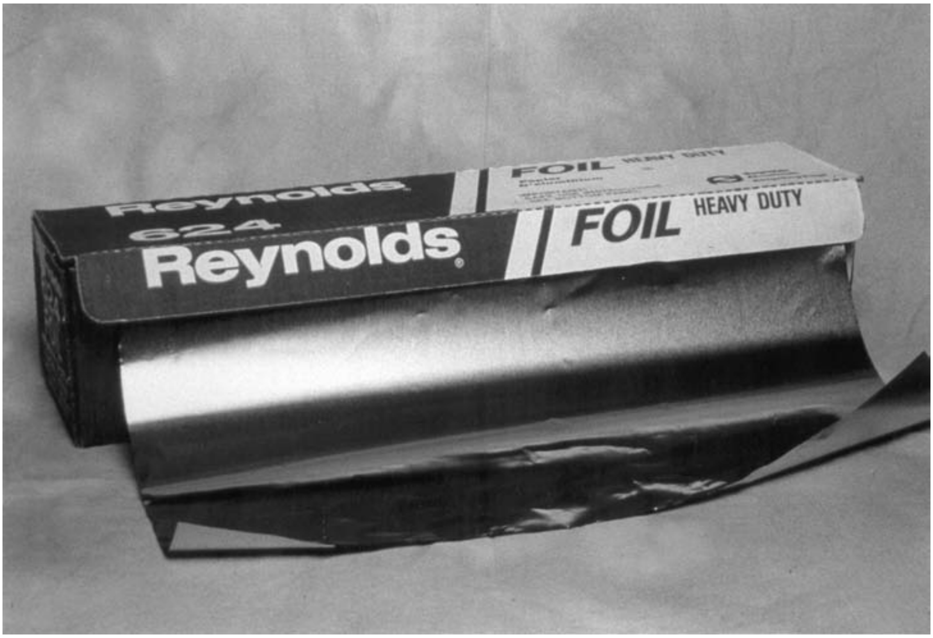

Fig. 1 Reynolds Heavy Duty Foil product, an example of aluminum food wrapping products made of various grades of 1xxx commercially pure aluminum.

2xxx: Al–Cu Alloys. The major characteristics of the 2xxx series are:

- Heat treatable
- High strength, at room and elevated temperatures
- Typical ultimate tensile strength range: 27–62 ksi
- Usually joined mechanically but some alloys are weldable

The 2xxx series are heat treatable and possess in individual alloys good combinations of high strength (especially at elevated temperatures), toughness, and, in specific cases, weldability. They are not as resistant to atmospheric corrosion as several other series and so are usually painted or clad for added protection.

The higher strength 2xxx alloys are widely used for aircraft (2024) and truck body (2014) applications, where they are generally used in bolted or riveted wing (Fig. 3) or fuselage (Fig. 4) construction. Some specific alloys in the series (e.g., 2219 and 2048) are readily joined by gas–metal arc (GMAW) or gas tungsten arc (GTAW) welding, and so are used for aerospace applications where that is the preferred joining method.

Alloy 2195 is a new Li-bearing aluminum alloy providing very high modulus of elasticity along with higher strength and comparable weldability to 2219 for space applications (Fig. 5).

For applications requiring very high strength plus high fracture toughness, there are high-toughness versions of several of the alloys (e.g., 2124, 2324, 2419) that have tighter control on the impurities that may diminish resistance to unstable fracture, all developed specifically for the aircraft industry.

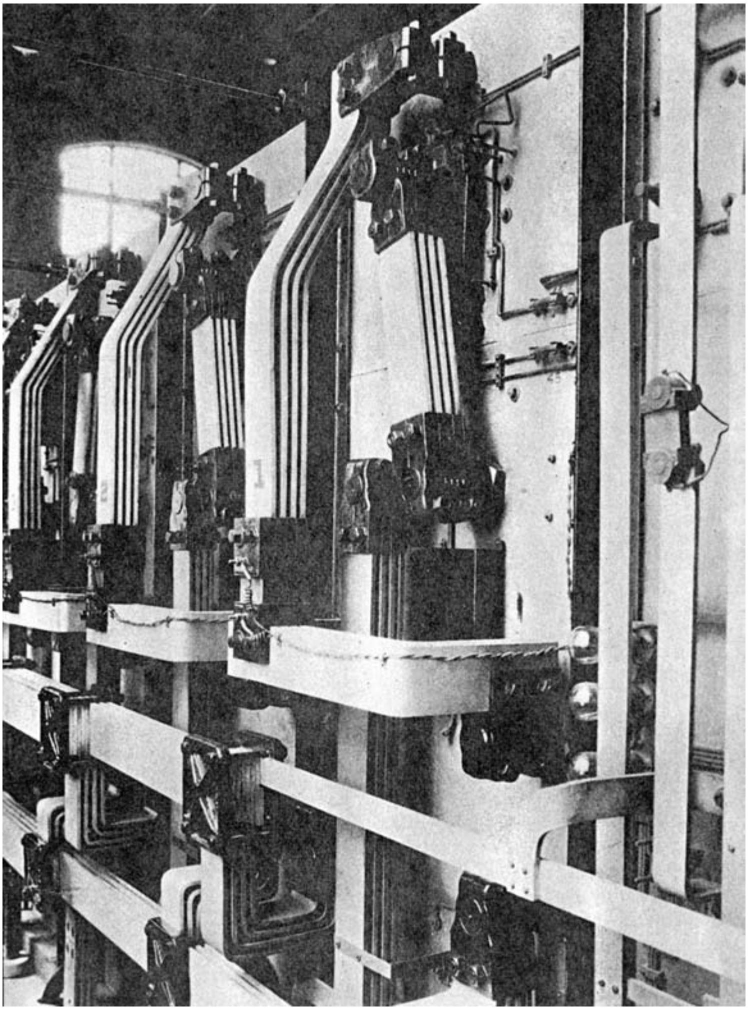

Fig. 2 Aluminum electrical bus bar installation with 1350 bus bar.

Alloys 2011, 2017, and 2117 are widely used for fasteners and screw-machine stock.

3xxx: Al–Mn Alloys. The major characteristics of the 3xxx series are:

- High formability and corrosion resistance with medium strength
- Typical ultimate tensile strength range: 16–41 ksi
- Readily joined by all commercial procedures

The 3xxx series are strain hardenable, have excellent corrosion resistance, and are readily welded, brazed, and soldered.

Alloy 3003 is widely used in cooking utensils and chemical equipment because of its superiority in handling many foods and chemicals, and in builders'

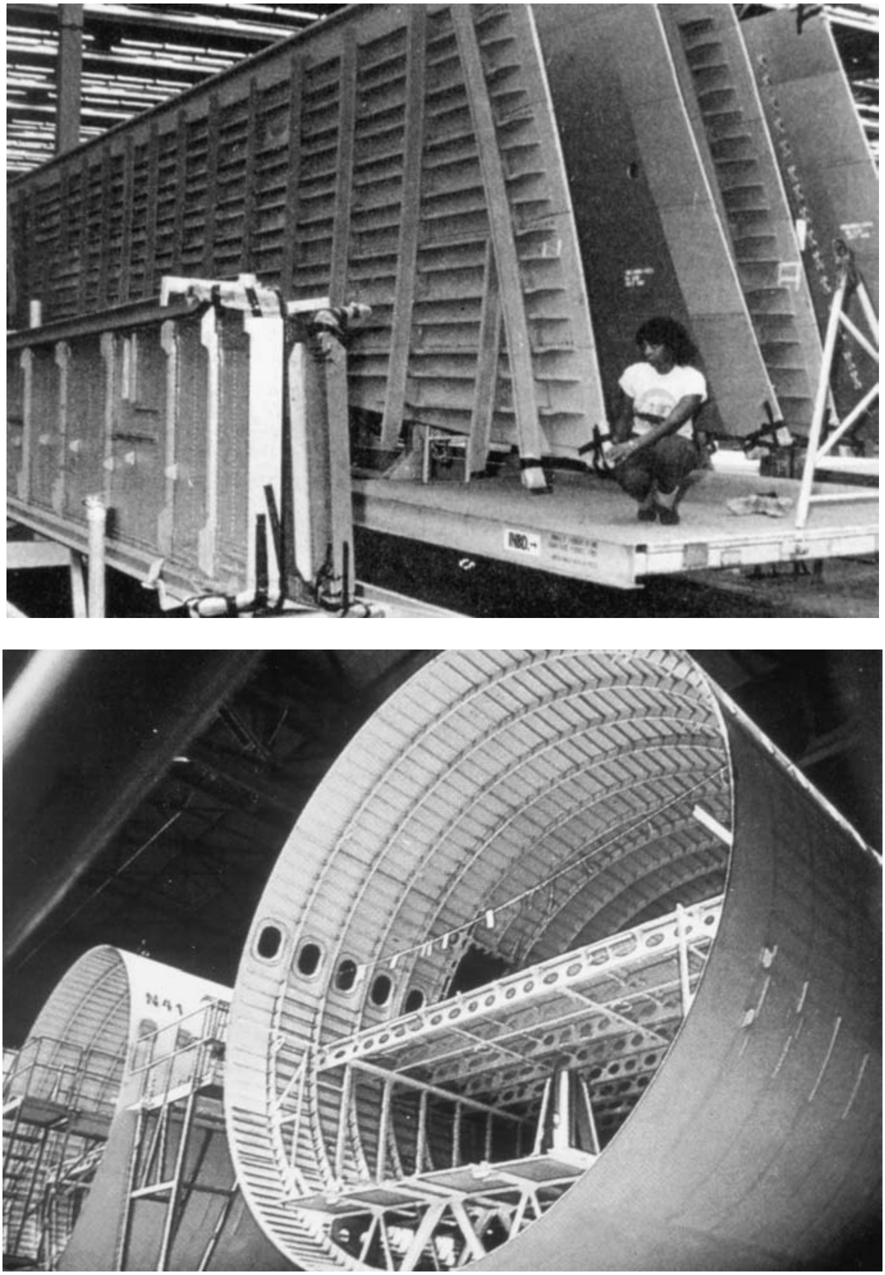

Fig. 3 Aircraft wing and fuselage structure includes extrusions and plate of 2xxx alloys like 2024, 2124, and 2618 and 7xxx alloys like 7050 and 7475. External sheet skin may be alclad 2024, 2524, 2618, or 7475; the higher purity cladding provides corrosion protection to the Al–Cu and Al–Zn–Mg alloys that may darken with age otherwise.

Fig. 4 Heavy dump and tank trucks and trailer trucks employ high-strength 2xxx or 6xxx extrusions for their structural members and tough 5xxx alloy sheet and plate for their load-carrying components.

hardware because of its superior corrosion resistance. Alloy 3105 is a principal for roofing and siding.

Because of the ease and flexibility of joining, 3003 and other members of the 3xxx series are widely used in sheet and tubular form for heat exchangers in vehicles and power plants (Fig. 6).

Alloy 3004 and its modification 3104 are the principals for the bodies of drawn and ironed can bodies for beverage cans for beer and soft drinks (Fig. 7). As a result, they are among the most used individual alloys in the aluminum system, in excess of 3.5 billion pounds per year.

4xxx: Al–Si Alloys. The major characteristics of the 4xxx series are:

- Heat treatable
- Good flow characteristics, medium strength

Fig. 5 Fuel tanks and booster rockets such as those of the Space Shuttle are 2xxx alloys, originally 2219 and 2419, now Al–Li "Weldalite" alloy 2195.

- Typical ultimate tensile strength range: 25–55 ksi
- Easily joined, especially by brazing and soldering

There are two major uses of the 4xxx series, both generated by their excellent flow characteristics provided by their relatively high silicon contents. The first is for forgings: the workhorse alloy is 4032, a medium high-strength, heat-treatable alloy used principally in applications such as forged aircraft pistons. The second major application is a weld filler alloy; here the workhorse is 4043, used for gas–metal arc (GMA) and gas–tungsten arc (GTA) welding 6xxx alloys for structural and automotive applications.

As noted, the same characteristic leads to both types of application: good flow characteristic provided by the high-silicon content. In the case of forgings, this ensures the complete and precise filling of complex dies; in the case of welding, it ensures complete filling of grooves in the members to be joined. For the same reason, other variations of the 4xxx alloys are used for the cladding on brazing sheet, the component that flows to complete the bond.

5xxx: Al–Mg Alloys. The major characteristics of the 5xxx series are:

- Strain hardenable
- Excellent corrosion resistance, toughness, weldability; moderate strength
- Building and construction, automotive, cryogenic, marine applications
- Typical ultimate tensile strength range: 18–51 ksi

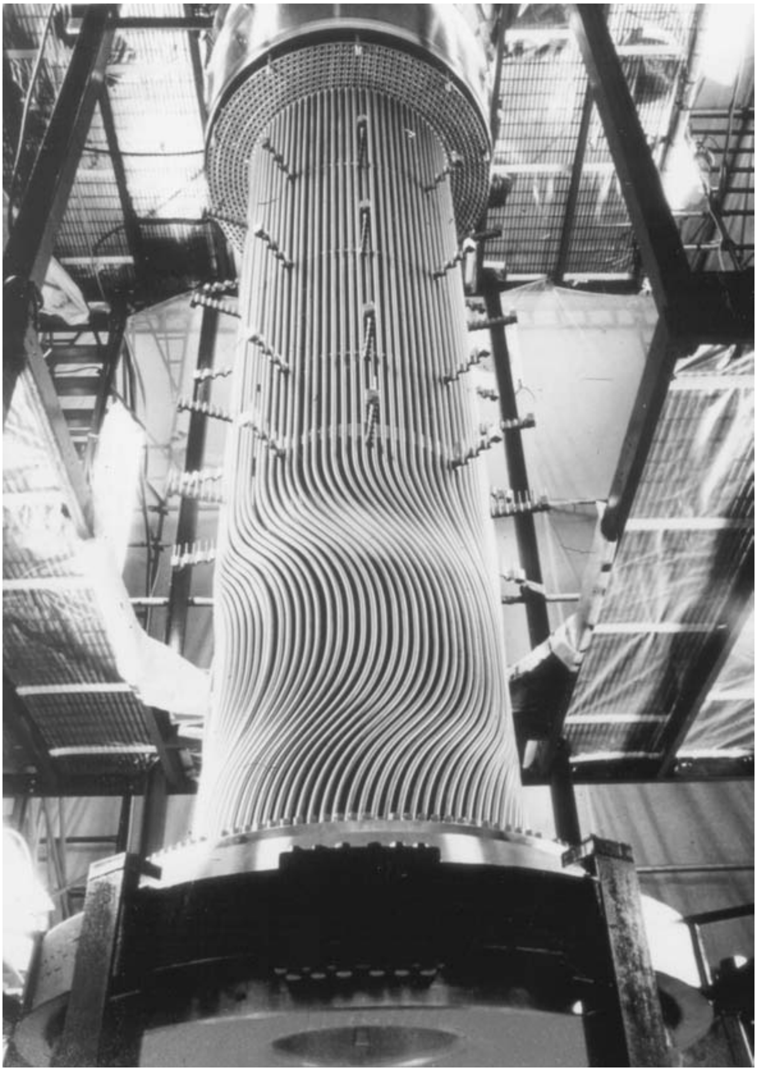

Fig. 6 Alloy 3003 tubing in large commercial power plant heat exchanger.

Al–Mg alloys of the 5xxx series are strain hardenable and have moderately high strength, excellent corrosion resistance even in saltwater, and very high toughness even at cryogenic temperatures to near absolute zero. They are readily welded by a variety of techniques, even at thicknesses up to 20 cm.

As a result, 5xxx alloys find wide application in building and construction (Fig. 8), highways structures including bridges (Fig. 9), storage tanks and pressure vessels, cryogenic tankage, and systems for temperatures as low as −459°F (−270°C, near absolute zero), transportation (Fig. 10), and marine applications (Fig. 11), including offshore drilling rigs (Fig. 12).

Alloys 5052, 5086, and 5083 are the workhorses from the structural standpoint, with increasingly higher strength associated with the increasingly higher

Fig. 7 Bodies of most beverage cans are alloy 3004, the ends are 5182, making it the largest volume alloy combination in the industry.

Mg content. Specialty alloys in the group include 5182, the beverage can end alloy (Fig. 7), and thus among the largest in tonnage; 5754 for automotive body frames and panels (Fig. 13); and 5252, 5457, and 5657 for bright trim applications, including automotive trim.

Care must be taken to avoid use of 5xxx alloys with more than 3% Mg content in applications where they receive continuous exposure to temperatures above 100°C (212°F). Such alloys may become sensitized and susceptible to stress corrosion cracking. For this reason, alloys such as 5454 and 5754 are recommended for applications where high-temperature exposure is likely.

6xxx: Al–Mg–Si Alloys. The major characteristics of the 6xxx series are:

- Heat treatable
- High corrosion resistance, excellent extrudibility; moderate strength
- Typical ultimate tensile strength range: 18–58 ksi
- Readily welded by GMAW and GTAW methods

The 6xxx alloys are heat treatable and have moderately high strength coupled with excellent corrosion resistance. A unique feature is their great extrudability, making it possible to produce in single shapes relatively complex architectural forms and also to design shapes that put the majority of the metal where it will most efficiently carry the highest tensile and compressive stresses (Fig. 14). This

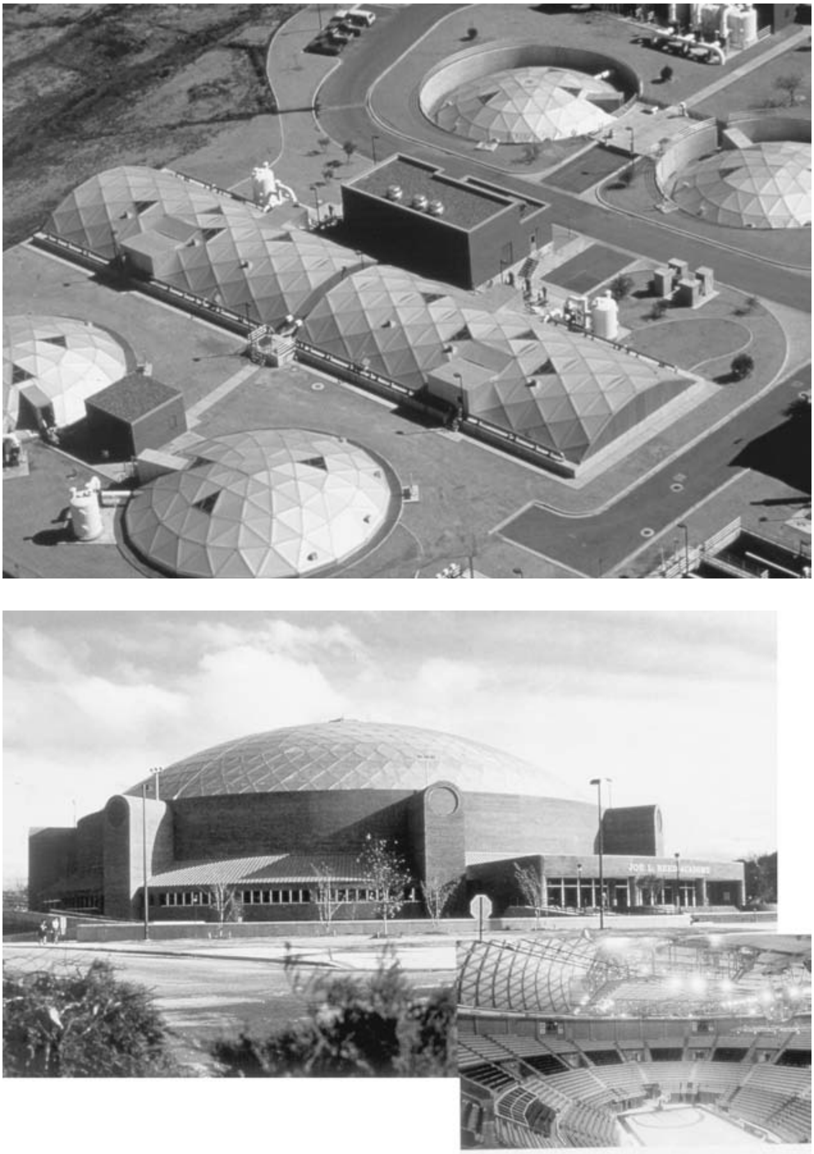

Fig. 8 Sheet of 5xxx alloys often forms the surface of geodesic dome structures, as in these examples of (*a*) a water treatment plant and (*b*) a wide-span arena roof. The structural supports are typically 6061 or 6063 extruded shapes or tubular members.

is a particularly important advantage for architectural and structural members where stiffness is important.

Alloy 6063 is perhaps the most widely used because of its extrudability; it is not only the first choice for many architectural and structural members, it has been the choice for the Audi automotive space frame members. A good example of its structural use was the all-aluminum bridge structure in Foresmo, Norway (Fig. 9); it was prefabricated in a shop and erected on the site in only a few days.

Fig. 9 The Foresmo bridge in northern Norway is an excellent example of the use of Al–Mg alloys for built-up girder systems; these photos illustrates a major advantage of replacement aluminum bridges: the ability to prefabricate the spans, (*a*) transport them, and (*b*) erect them in place quickly, minimizing the disruption to traffic.

Higher strength alloy 6061 extrusions and plate find broad use in welded structural members such as automotive, truck (Fig. 4), and marine frames, railroad cars, and pipelines. Alloys like 6111 provide a fine combination of strength and formability, useful for external automotive panels (Fig. 13*b*).

Among specialty alloys in the series: 6066-T6, with high strength for forgings; 6071 for the highest strength available in 6xxx extrusions; and 6101 and 6201 for high-strength electrical bus and electrical conductor wire, respectively.

Fig. 10 Alloy 5454 has been widely used for railcar body construction where heavy loads, such as coal, and potentially high temperatures, may be involved.

7xxx: Al–Zn Alloys. The major characteristics of the 7xxx series are:

- Heat treatable
- Very high strength; special high toughness versions
- Typical ultimate tensile strength range: 32–88 ksi
- Mechanically joined

The 7xxx alloys are heat treatable and, among the Al–Zn–Mg–Cu versions in particular, provide the highest strengths of all aluminum alloys. These alloys are not considered weldable by commercial processes, and are regularly used with riveted construction.

The widest application of the 7xxx alloys has historically been in the aircraft industry (Fig. 3), where fracture-critical design concepts have provided the impetus for the high-toughness alloy development. There are several alloys in the series that are produced especially for their high toughness, notably 7050, 7150, 7175, and 7475; for these alloys, controlled impurity levels, particularly of Fe and Si, maximize the combination of strength and fracture toughness. Forgings of these alloys (Fig. 15) are often used for large structural members in aircraft.

The high-strength/density combination for 7075-T73 (and 2014-T6) have made them choices for drill pipe where the long lengths needed for deep wells require lightweight alloys.

The atmospheric corrosion resistance of the 7xxx alloys is not as high as that of the 5xxx and 6xxx alloys, so in such service they are usually coated or, for sheet and plate, used in an alclad version. Also, special tempers have been developed to improve their resistance to exfoliation and stress–corrosion cracking, the T76 and T73 types, respectively. These tempers are especially recom-

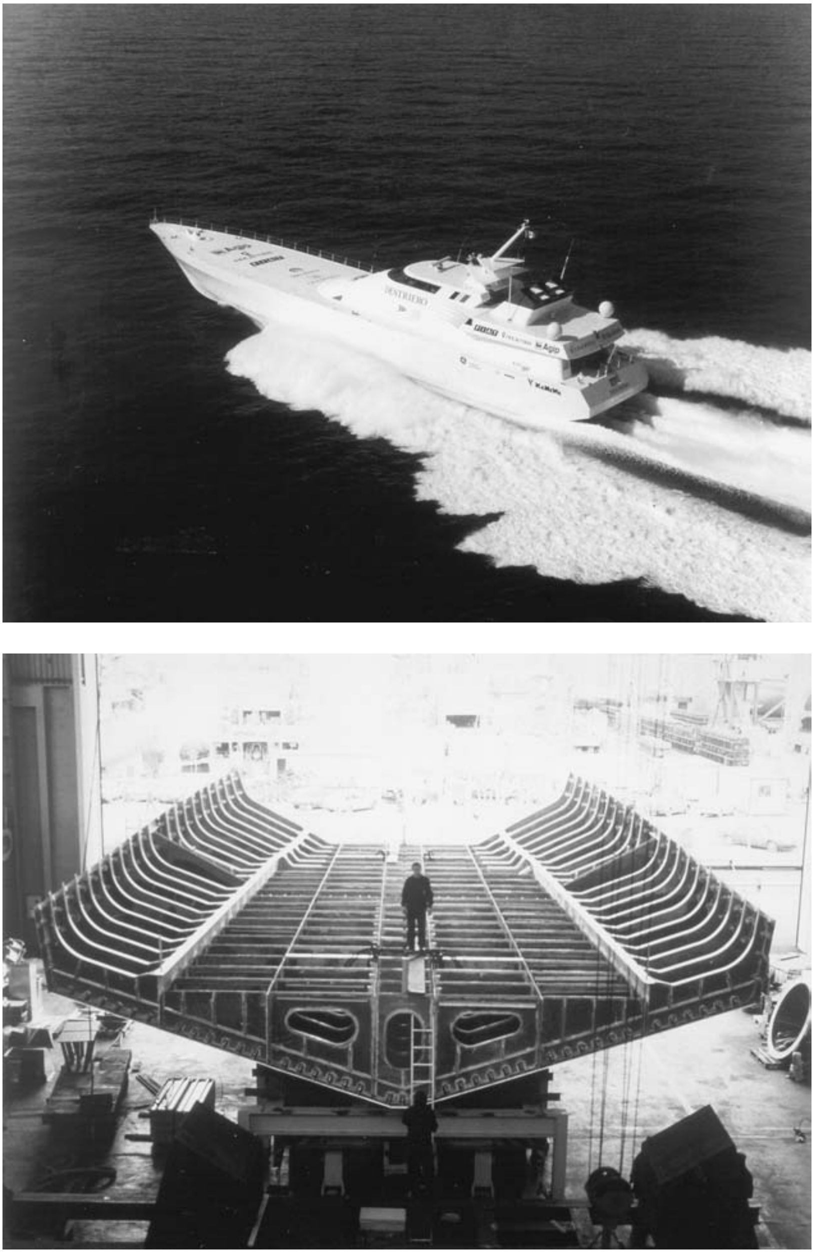

Fig. 11 High-speed single-hull ships (*a*) like the *Proserio* employ 5083 or 5383-H113/H321 machined plate for hulls, (*b*) internal hull stiffeners, decking, and superstructure.

Fig. 12 Demands of the superstructures of offshore oil rigs in high humidity and water exposure are met with 5454, 5086, and 5083 Al–Mg alloy welded construction.

mended in situations where there may be high short transverse (through the thickness) stresses present during exposure to atmospheric or more severe environments.

The Cu-free 7xxx alloys have lower strength but are tougher and are both readily extrudable and weldable, so alloys like 7005 and 7029 find their way into applications like guard rail and automotive and truck bumpers.

8xxx: Alloys with Al + Other Elements (Not Covered by Other Series). The major characteristics of the 8xxx series are:

- Heat treatable
- High conductivity, strength, hardness
- Typical ultimate tensile strength range: 17–35 ksi

The 8xxx series is used for those alloys with lesser-used alloying elements such as Fe, Ni, and Li. Each is used for the particular characteristics it provides the alloys.

Fe and Ni provide strength with little loss in electrical conductivity and so are used in a series of alloys represented by 8017 for conductors.

Lithium in alloy 8090 provides exceptionally high strength and modulus, and so this alloy is used for aerospace applications where increases in stiffness combined with high strength reduces component weight.

Cast Alloys

In comparison with wrought alloys, casting alloys contain larger proportions of alloying elements such as silicon and copper, This results in a largely hetero-

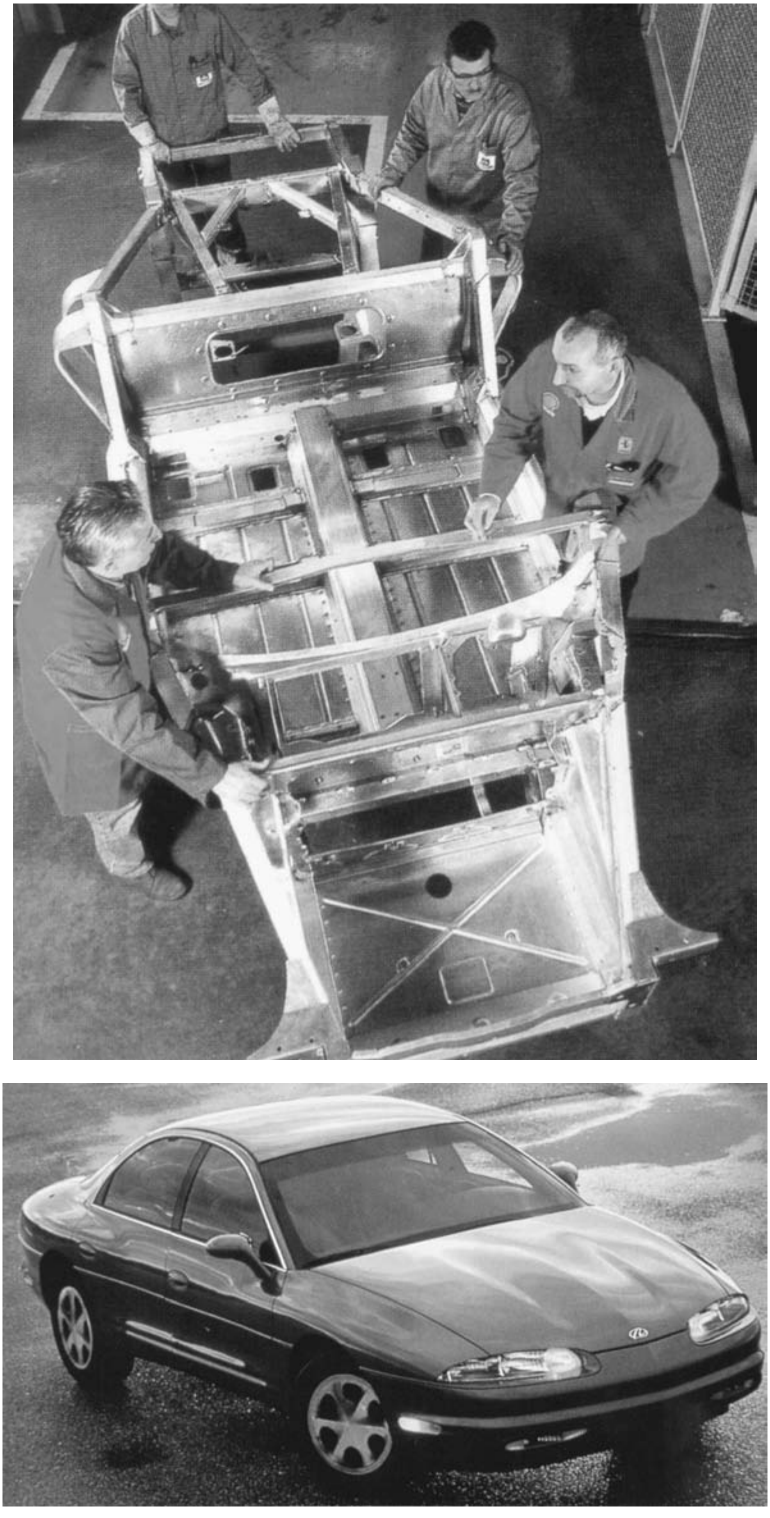

Fig. 13 Automotive structures are likely to employ increasing amounts of 5754-0 formed sheet for parts such as internal door stiffeners or (*a*) the entire body in white; (*b*) external body panels are more likely to be higher strength 6111-T4.

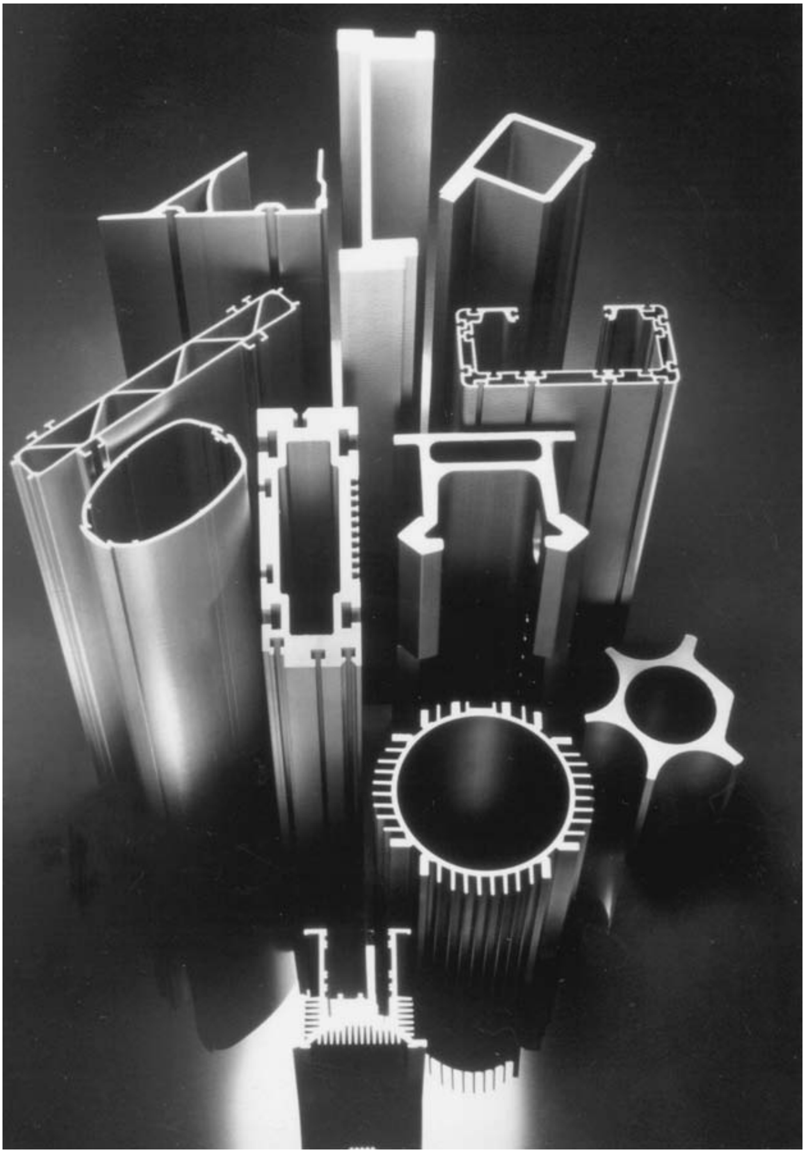

Fig. 14 Power of extruded Al–Mg–Si alloys is the "put the metal where you need it" flexibility these alloys and the extrusion process provide.

geneous cast structure, i.e., one having a substantial volume of second phases. This second phase material warrants careful study since any coarse, sharp, and/or brittle constituent can create harmful internal notches and nucleate cracks when the component is later put under load. Fatigue performance is very sensitive to large heterogeneities, especially at or near the surface. As will be shown later, good metallurgical and foundry practice can largely prevent such defects.

The elongation and strength, especially in fatigue, of most cast products are relatively lower than those of wrought products. This is because current casting

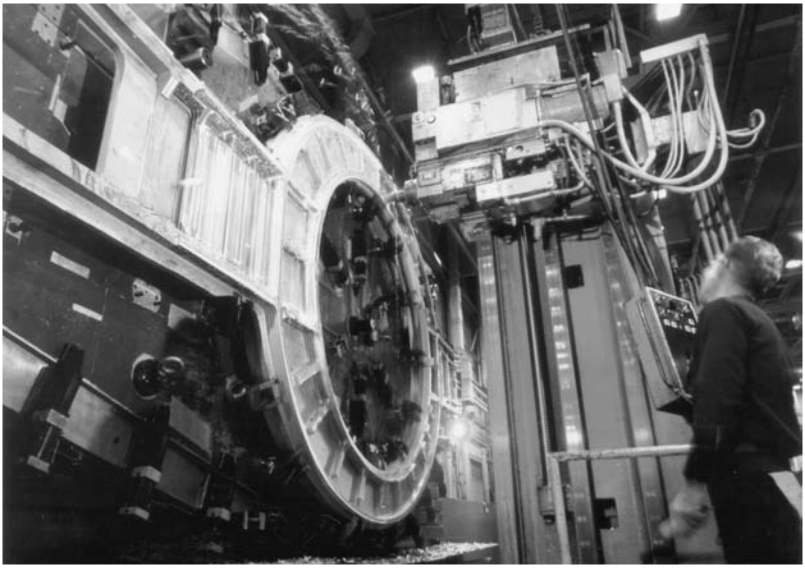

Fig. 15 Example of a premium forged aircraft part, usually of alloys such as 7050 or 7175-T74.

practice is as yet unable to reliably prevent casting defects. In recent years, however, innovations in casting processes such as squeeze casting have brought about some significant improvement in the consistency and level of properties of castings, and these should be taken into account in selecting casting processes for critical applications.

For applications where high ductility and toughness along with high strength, relatively high-purity versions of casting alloys like A356.0-T6 (rather than 356.0-T6) and A357.0-T6 (rather than 357.0-T6) are recommended.

2xx.x: Al–Cu Alloys. The major characteristics of the 2xx.x series are:

- Heat treatable; sand and permanent mold castings
- High strength at room and elevated temperatures; some high toughness alloys
- Approximate ultimate tensile strength range: 19–65 ksi

The strongest of the common casting alloys are heat-treated 201.0 and 204.0, which have found important application in the aerospace industry. Its castability is somewhat limited by a tendency to microporosity and hot tearing, so that it is best suited to investment casting. Its high toughness makes it particularly suitable for highly stressed components in machine tool construction, in electrical engineering (pressurized switchgear casings), and in aircraft construction.

Besides the standard aluminum casting alloys, there are special alloys for particular components, for instance, for engine piston heads, integral engine blocks, or bearings. For these applications the chosen alloy needs good wear resistance and a low friction coefficient, as well as adequate strength at elevated service temperatures. A good example is the alloy 203.0, which to date is the aluminum casting alloy with the highest strength at around 400°F (200°C).

3xx.x: Al–Si + Cu or Mg Alloys. The major characteristics of the 3xx.x series are:

- Heat treatable; sand, permanent mold, and die castings
- Excellent fluidity; high strength/some high-toughness alloys
- Approximate ultimate tensile strength range: 19–40 ksi
- Readily welded

The 3xx.x series of castings are one of the most widely used because of the flexibility provided by the high-silicon contents and its contribution to fluidity plus their response to heat treatment which provides a variety of high-strength options. As a result, they are the best choice for large and complex castings (Fig. 16). Further the 3xx.x series may be cast by a variety of techniques ranging from relatively simple sand or die casting to very intricate permanent mold, investment castings, and the newer thixocasting and squeeze casting technologies (Fig. 17).

Among the workhorse alloys are 319.0 and 356.0/A356.0 for sand and permanent mold casting, with A356.0-T6 for such critical applications as automotive wheels (Fig. 18). For die castings, 360.0, 380.0/A380.0, and 390.0 are the most widely used. The newer squeeze/forge cast technologies have generally employed A356.0-T6 and A357.0-T6.

Alloy 332.0 is also one of the most frequently used aluminum casting alloys because it can be made almost exclusively from recycled scrap.

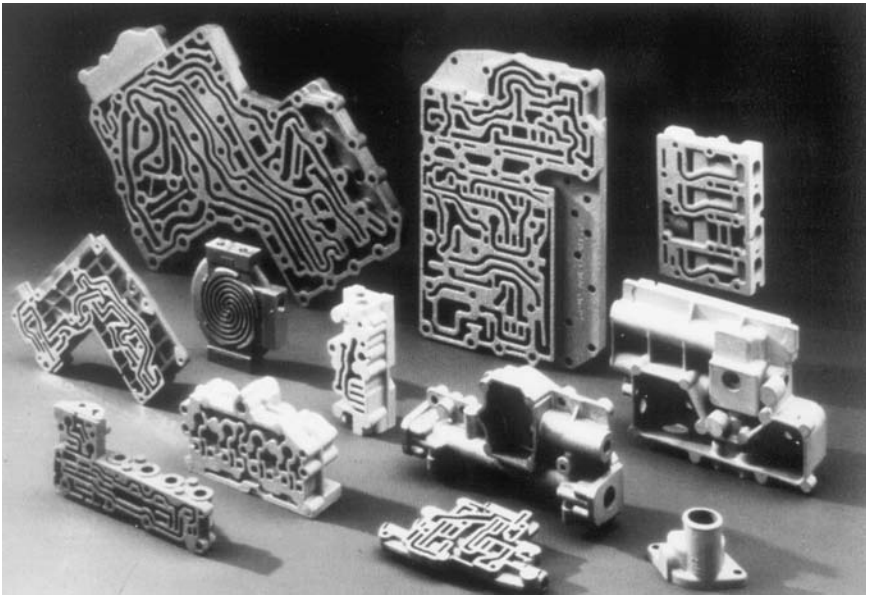

Fig. 16 Complex 3xx.x castings made by the investment casting processes provide the ability to obtain exceptionally intricate detail and fine quality.

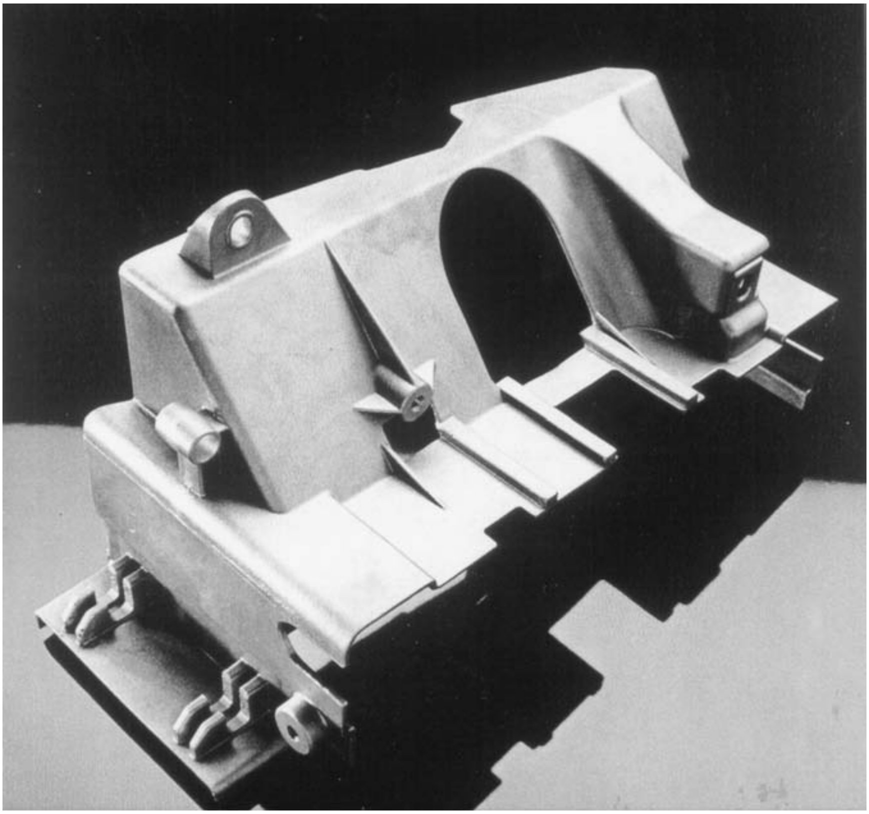

Fig. 17 Thixoformed A356.0-T6 and A357.0-T6 may be used for critical aircraft components.

Fig. 18 Automotive wheels are often cast A356.0-T6.

4xx.x: Al–Si Alloys. The major characteristics of the 4xx.x series are:

- Nonheat treatable; sand, permanent mold, and die castings
- Excellent fluidity; good for intricate castings
- Approximate ultimate tensile strength range: 17–25 ksi

Alloy B413.0 is notable for its very good castability and excellent weldability, which are due to its eutectic composition and low melting point of 700°C. It combines moderate strength with high elongation before rupture and good corrosion resistance. The alloy is particularly suitable for intricate, thin-walled, leakproof, fatigue-resistant castings.

These alloys have found applications in relatively complex cast parts for typewriter and computer housings and dental equipment, and also for fairly critical components in marine and architectural applications.

5xx.x: Al–Mg Alloys. The major characteristics of the 5xx.x series are:

- Nonheat treatable; sand, permanent mold, and die
- Tougher to cast; provides good finishing characteristics
- Excellent corrosion resistance, machinability, surface appearance
- Approximate ultimate tensile strength range: 17–25 ksi

The common feature of this group of alloys is good resistance to corrosion. Alloys 512.0 and 514.0 have medium strength and good elongation and are suitable for components exposed to seawater or to other similar corrosive environments. These alloys are often used for door and window fittings, which can be decoratively anodized to give a metallic finish or in a wide range of colors. Their castability is inferior to that of the AI–Si alloys because of its magnesium content and consequently long freezing range. For this reason it tends to be replaced by 355.0/AJSi5Mg, which has long been used for similar applications.

For die castings where decorative anodizing is particularly important, alloy 520.0 is quite suitable.

7xx.x: Al–Zn Alloys. The major characteristics of the 7xx.x series are:

- Heat treatable; sand and permanent mold cast (harder to cast)
- Excellent machinability and appearance
- Approximate ultimate tensile strength range: 30–55 ksi

Because of the increased difficulty in casting 7xx.x alloys, they tend to be used only where the excellent finishing characteristics and machinability are important. Representative application include furniture, garden tools, office machines, and farming and mining equipment

8xx.x: Al–Sn Alloys. The major characteristics of the 8xx.x series are:

- Heat treatable; sand and permanent mold castings (harder to cast)
- Excellent machinability

- Bearings and bushings of all types
- Approximate ultimate tensile strength range: 15–30 ksi

Like the 7xx.x alloys, 8xx.x alloys are relatively hard to cast and tend to be used only where their combination of superior surface finish and relative hardness are important. The prime example is for parts requiring extensive machining and for bushings and bearings.

4.2 Applications by Market Area

In the paragraphs that follow, a review will be made of the alloys often selected for products in a number of the major markets in which aluminum is used.

Electrical Markets

The major products for which aluminum is used in electrical applications are electric cable and bus conductor, where the high electrical conductivity (60% IACS) makes aluminum a cost-effective replacement for copper products:

- Electrical conductor wire—1350 where no special strength requirements exist and 6201 where a combination of high strength and high conductivity are needed.
- Bus conductor—6101 (Fig. 2)
- Electrical cable towers—6063 or 6061 extruded shapes

Building and Construction Markets

Building and construction encompasses those markets where architectural and/or structural requirements come together. Such applications include residential housing, commercial store fronts and structures, conference centers and areas (i.e., long roof bay requirements), highway bridges and roadside structures, and a variety of holding tanks and chemical structures (also considered under Petroleum and Chemical Industry Components). Among the choices:

- Bridges and other highway structures—6063 and 6061 extrusions (Figs. 8, 9); 5083, 5083, and 5454 plate (Fig. 9)
- Storefronts, curtain wall—6063 extrusions
- Building sheet; siding—3005, 3105; 5005 sheet
- Arena and convention center roofs—6061 extrusions with 5xxx alloy sheet panels (Fig. 8)
- Residential housing structures—6063 extrusions
- Architectural trim—5257, 5657, and 6463
- Composite wall panels—5xxx alloy sheet plus expanded polymers

Automobile, Van, SUV, Bus, and Truck Applications

Automotive structures require a combination of aluminum castings, sheet and extrusions to cover all good opportunities to increase gasoline mileage and reduce pollutants. Among examples are the following:

- Frame—5182 or 5754 sheet (Fig. 13*a*) or, for space frame designs, 6063 or 6061 extrusions
- External body sheet panels where dent resistance is important—2008 and 6111(Fig. 13*b*)
- Inner body panels—5083 and 5754
- Bumpers—7029 and 7129
- Air conditioner tubes, heat exchangers—3003 (Fig. 6)
- Auto trim—5257, 5657, and 5757
- Door beams, seat tracks, racks, rails, etc.—6061 or 6063
- Hood, decklids—2036, 6016, and 6111
- Truck beams—2014 and 6070 (Fig. 4)
- Truck trailer bodies—5454, 5083, and 5456 (Fig. 4)
- Wheels—A356.0 (Fig. 18), formed 5xxx sheet, or forged 2014-T6
- Housings, gear boxes—357.0 and A357.0 (Fig. 16)

Aircraft and Aerospace Applications

Aircraft and aerospace applications require high strength combined with, depending upon the specific component, high fracture toughness, high corrosion resistance, and/or high modulus (sometimes all three). The result has been a great number of alloys and tempers developed specifically for this market, as illustrated by the examples below:

- Space mirror—high-purity aluminum
- Wing and fuselage skin—2024, alclad 2024, and 7050 plate or extrusions (Fig. 3)
- Wing structures—2024, 2124, 2314, and 7050 stiffened extrusions (Fig. 3)
- Bulkhead—2197, 7049, 7050, and 7175
- Rocket tankage—2195, 2219, and 2419 (Fig. 5)
- Engine components—2618
- Propellers—2025
- Rivets—2117, 6053
- If high modulus is critical—Li-bearing alloys 2090, 2091, 2195, or 8090
- If high fracture toughness is critical—2124, 2324, 7050, 7175, and 7475
- For maximum fracture toughness—7475
- If stress–corrosion resistance is important—7X50 or 7X75 in the T73-type temper
- If resistance to exfoliation attack is vital—7xxx alloys in the T76-type temper
- For welded construction, as for shuttle tanks—5456, 2219, and 2195

Marine Transportation

Many aluminum alloys readily withstand the corrosive attack of marine saltwater, and so find applications in boats, ships, offshore stations, and other components that are immersed in saltwater:

- Hull material—5083, 5383, 6063 and 6061 (Fig. 11)
- Superstructure—5083 and 5456
- Structural beams—5083, 5383, 6063, and 6061 (Fig. 11)
- Off-shore stations, tanks—5083 and 5456 (Fig. 12)

Rail Transportation

Much as for auto and truck bodies, aluminum lends itself to railcar structural and exterior panel applications:

- Beams—2014, 6061, and 6070
- Exterior panels—5456 and 6111
- Tank cars—5454 and 5083
- Coal cars—5454, 5083, and 5456 (Fig. 10)
- Cars for hot cargo—5454

Packaging Applications

Packaging applications require either great ductility and corrosion resistance for foil and wrapping applications or great strength and workability for rigid container sheet applications, i.e., cans. Alloy choices include:

- Aluminum foil for foods—1175 (Fig. 1)
- Rigid container (can) bodies—3004 (Fig. 7)
- Rigid container (can) ends—5182

Petroleum and Chemical Industry Components

The excellent combination of high strength combined with superior corrosion resistance plus weldability makes a number of aluminum alloys ideal for chemical industry applications, even some involving very corrosive fluids:

- Chemical piping—1060, 5254, and 6063
- Pressure vessels (ASME code)—5083, 5086, 6061, and 6063
- Pipelines—6061, 6063, and 6070
- Cryogenic tankage—5052, 5083, 5454, 6063, and 6061
- Containers for hydrogen peroxide—5254 and 5652

Other Markets

While not major markets in themselves, a variety of specialty products find great advantage in aluminum alloys:

- Screw machine products—2011 and 6262
- Appliances—5005 and 5052
- Tread plate—6061
- Weld wire—4043 (for welding 6xxx alloys), 5356, 5183, and 5556 (for welding 5xxx alloys)

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CHAPTER 5

COPPER AND COPPER ALLOYS

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1 INTRODUCTION

Copper and copper alloys comprise one of the broadest and most versatile groups of engineering materials. Almost 500 copper alloys are currently recognized in the United States, and hundreds more are classified under international standards. Copper alloys are also produced in all common product forms, further expanding the possible choices.

The large selection may seem daunting, but choosing the correct alloy is simplified by the fact that copper metals are normally chosen for particular physical or mechanical properties, and alloys with the desired properties can easily be sorted out. A need for very high electrical or thermal conductivity, for example, generally points to one of the coppers or high-copper alloys, while superior machinability suggests that a leaded alloy ought to be considered. There are likewise alloys that are selected primarily for their superior corrosion resistance, formability, castability, mechanical properties, biofouling resistance, and biostatic behavior, among other things.

On the other hand, the main reason copper and it alloys are so widely used is that they offer a better *combination* of useful properties than can be found in

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Handbook of Materials Selection. Edited by Myer Kutz

other materials. This fortunate circumstance reduces the level of compromise needed to satisfy all design requirements satisfactorily.

The designer's first choice, however, is usually that of a suitable product form: wire or rod; sheet, strip, or plate; castings; and so forth, and for that reason, alloys listed in this chapter are grouped according to the forms in which those alloys are customarily produced. Data is presented for a broad and representative selection of alloys. Unfortunately, space constraints prohibit inclusion of data for all currently registered alloys in all product forms and in all tempers. Comprehensive information on all alloys is available on line at *The Copper Page* (http://www.copper.org), through the Copper Data Center (http://www.csa.com/copperdata/), and from the Copper Development Association (CDA).

2 STRUCTURE OF THE COPPER INDUSTRY

Copper is truly a global metal, found in economic quantities in all continents except Antarctica. Chile currently leads the world in copper production, having passed the United States (which retains second place) in the early 1990s. Other major copper-producing countries include Peru, Canada, Mexico, Poland, Zaire, Zambia, Australia, Philippines, Indonesia, Papua New Guinea, and the Commonwealth of Independent States (CIS; former Soviet Union). The (former) U.S. Bureau of Mines estimates that worldwide land-based copper resources stand at 1.6 billion metric tons (mt), with an additional 1.8 billion mt contained in deep-sea nodules. Global copper depletion, once forecast as a looming threat, is now seen as an unfounded concern.

Not all countries that mine substantial quantities of copper actually produce commercial metal. Copper concentrates and an impure form of metal known as blister copper are traded worldwide to countries having the smelters and refineries needed to complete the metal production process. Western Europe and Japan, for example, mine little copper but produce significant amounts for their own use and for export.

Copper is a commodity metal, and the Comex and the London Metal Exchange fix published prices. The exchanges account for only a fraction of the copper actually traded worldwide, however, and the bulk of trading takes place between producers and users.

Major U.S. copper-mining states include Arizona, Utah, and Nevada plus, to a lesser extent, New Mexico. Large deposits in the Upper Peninsula of Michigan, once the primary source of domestic copper, have largely been mined out. The U.S. copper-mining industry has consolidated considerably in recent years and only three domestic producers—Phelps Dodge, Asarco Grupo Mexico, and Kennecott Utah Copper—can be considered major players in the sense that they operate large mines on U.S. soil. On the other hand, these companies and others have extensive holdings in copper-producing regions elsewhere in the world, and, conversely, a number of foreign companies either have interests in, or wholly own U.S.-based copper producers, including two of those named above.

Copper smelters, which produce blister copper from concentrates, are generally located near mines. Refineries, where blister is purified to 99.9+% pure cathode copper through an electrolytic process, are somewhat more widespread, although most are also found in the copper-mining states. Several large refineries are located in Texas.

Copper products are produced by fabricators: wire mills, brass mills, foundries, and metal powder plants. Their plants are found throughout the country. The U.S. copper industry was once very much vertically integrated, but it has become increasingly diversified and few fabricators now retain formal ties to producers.

As explained below, copper wire and cable and other electrical products are usually made from newly mined and refined copper. Copper tube and alloy products are made from a combination of refined and scrap copper, as well as copper alloy scrap. Copper is, in fact, among the most thoroughly recycled industrial metals.

3 COPPER ALLOY DESIGNATIONS

In the United States and several other countries, copper and copper alloys are classified under the Unified Numbering System for Metals and Alloys (UNS), a system managed jointly by the American Society for Testing and Materials (ASTM) and the Society of Automotive Engineers (SAE). CDA is responsible for assigning numbers to copper alloys, which are identified by a five-digit code preceded by the letter "C." The codes are extensions of a previous and once widely used three-digit numbering system, which was also administered by CDA. Thus, for example, the alloy formerly known as copper alloy 360 by its three-digit "CDA number" is now designated UNS C36000. It should be noted that the UNS numbers are not specifications; they are merely standard designations for defined compositions.

Copper alloys are sometimes also referred to by descriptive historical names such as "free-cutting brass" and "naval brass." Colorful as such names may be, they can be ambiguous, and it is strongly recommended that alloys be called out by their UNS numbers.

In addition to its UNS number, specification of a copper or copper alloy usually requires inclusion of a *temper designation*. Temper is a term that identifies the metallurgical state of an alloy as well as the mechanical and physical properties resulting from its processing history. Terms such as annealed, half-hard, and precipitation heat treated, along with their coded abbreviations, are listed in Tables 1 and 2 for wrought and cast alloys, respectively. The tables are taken from ASTM B601. Mechanical property data tables in this chapter include the most commonly specified tempers for the alloys listed. Data corresponding to tempers not listed here may be found at http://properties.copper.org/ and in publications available from CDA.

Copper Alloy Families. The copper metals are conventionally grouped into several families according to similarities in composition, and hence, properties. UNS numbers from C10100 through C79999 denote wrought (i.e., drawn, rolled, extruded, or forged) alloys. Cast alloys are numbered from C80100 through C99999.

The family identified as *coppers* include metals that have a designated minimum copper content of 99.3% or higher. Wrought coppers (wire, rolled products, extrusions, etc.) include UNS designations ranging from C10100 through C15999, although all numbers are not in use. Cast coppers are numbered from

Table 1 Standard Temper Designations for Wrought Coppers and Copper Alloys
(Based on ASTM B601)

Annealed Tempers—O	Temper Names
O25	Hot rolled and annealed
O30	Hot extruded and annealed
O50	Light anneal
O60	Soft anneal
O61	Annealed (also mill annealed)
O70	Dead soft anneal

Annealed Tempers, with Grain Size Prescribed—OS	Nominal Average Grain Size (mm)
OS005	0.005
OS010	0.010
OS015	0.015
OS020	0.020
OS025	0.025
OS035	0.035
OS050	0.050
OS060	0.060
OS070	0.070
OS100	0.100
OS120	0.120
OS150	0.150
OS200	0.200

Cold-Worked Tempers Based on Cold Rolling or Drawing—H	Temper Names
H00	⅛ hard
H01	¼ hard
H02	½ hard
H03	¾ hard
H05	Hard
H06	Extra hard
H08	Spring
H10	Extra spring
H12	Special spring
H13	Ultra spring
H14	Super spring

Cold-Worked Tempers Based on Particular Products (Wire)—H	Temper Names
H60	Cold heading, forming
H63	Rivet
H64	Screw
H66	Bolt
H70	Bending
H80	Hard drawn

Cold Worked and Stress Relieved—HR	Temper Names
HR01	¼ Hard and stress relieved
HR02	½ Hard and stress relieved
HR04	Hard and stress relieved
HR08	Spring and stress relieved
HR10	Extra spring and stress relieved

Cold-Worked Tempers with Added Treatments—HR	Temper Names
HR50	Drawn and stress relieved

As-Manufactured Tempers—M	Temper Names
M10	As hot forged—air cooled
M11	As hot forged—quenched
M20	As hot rolled
M30	As extruded

Solution Heat Treated—TB	Temper Name
TB00	Solution heat treated (A)

Solution Treated and Cold Worked—TD	Temper Names
TD00	Solution heat treated and cold worked: ⅛ hard
TD01	Solution heat treated and cold worked: ¼ hard
TD02	Solution heat treated and cold worked: ½ hard
TD03	Solution heat treated and cold worked: ¾ hard
TD04	Solution heat treated and cold worked: hard (H)

Solution Heat Treated and Precipitation Heat Treated—TF	Temper Names
TF00	Solution heat treated and aged (AT)

Quench Hardened—TQ	Temper Names
TQ00	Quench hardened
TQ30	Quench hardened and tempered

Solution Heat Treated, Cold Worked and Precipitation Heat Treated—TH	Temper Names
TH01	¼ Hard and precipitation heat treated (¼HT)
TH02	½ Hard and precipitation heat treated (½HT)
TH03	¾ Hard and preciptiation heat treated (¾HT)
TH04	Hard and precipitation heat treated (HT)

Mill Hardened Tempers—TM	Manufacturing Designation
TM00	AM
TM01	¼ HM
TM02	½ HM
TM04	HM
TM06	XHM
TM08	XHMS

Table 2 Temper Designations for Coppers Casting Alloys
(Based on ASTM B601)

Temper Designations	Temper Names
	Annealed—O
O10	Cast and annealed (homogenized)
O11	As-cast and precipitation heat treated
	As-Manufactured—M
M01	As sand cast
M02	As centrifugal cast
M03	As plaster cast
M04	As pressure die cast
M05	As permanent mold cast
M06	As investment cast
M07	As continuous cast
	Heat-Treated—TQ
TQ00	Quench hardened
TQ30	Quench hardened and tempered
TQ50	Quench hardened and temper annealed
	Solution Heat Treated and Spinodal Heat Treated—TX
TX00	Spinodal hardened (AT)
	Solution Heat Treated—TB
TB00	Solution heat treated (A)
	Solution Heat Treated and Precipitation Heat Treated—TF
TF00	Precipitation hardened (AT)

C80100 through C81399. These metals comprise various grades of commercially pure copper that are used primarily for electrical and electronic products.

High-copper alloys include those wrought grades with designated copper contents less than 99.3% but higher than 96.0% (UNS C16200 through C19999), which do not fall into any other copper alloy group. Cast high-copper alloys (C81300 through C82800) have minimum copper contents in excess of 94.0% and may contain silver for special purposes. Wrought or cast, these alloys combine electrical and thermal conductivities approaching those of pure copper but with higher strength, hardness, and wear resistance. Corrosion resistance may be higher or lower than that exhibited by the pure metal, depending on interactions between the environment and specific alloys. Common uses for high-copper alloys include electrical connectors, resistance welding electrodes, slip rings, coax cable shields, and trolley wire.

Brasses are alloys containing zinc as the principal alloying element with or without secondary alloying elements such as iron, aluminum, nickel, and silicon. The wrought brasses comprise three subfamilies:

- Copper–zinc alloys (ordinary brasses), C21000 through C28999
- Copper–zinc–lead alloys (leaded brasses), C30000 through C39999
- Copper–zinc–tin alloys (tin brasses), C40000 through C9999

Cast brasses are grouped into four subfamilies:

- Copper–tin–zinc alloys (including semired brasses and leaded red brasses, C83300 through C83999, semired and leaded semired brasses, C84000 through C84999, and yellow and leaded yellow brasses, C85000 through C85999)
- So-called manganese bronze alloys (also known as high-strength yellow brasses) and their leaded variants, C86000 through C86999
- Copper–zinc–silicon alloys (silicon brasses and "bronzes"), C87000 through C87999

This large group of alloys offers a wide range of useful properties, which vary widely according to composition, product form, and temper. Conductivity, while substantially lower than copper or high-copper alloys, is often adequate for electrical applications such as connectors. Various brasses, again depending on composition, exhibit superior formability, machinability, and/or castability, as well as good corrosion resistance and moderate to high mechanical properties. Among the economical yellow brasses are wrought grades with excellent hot forging properties and cast varieties that are well suited for the cost-effective permanent mold (gravity die) casting process, described below. Applications for wrought brasses include springs, contacts, connectors, welding wire, jewelry, munitions, tube sheets, marine hardware, architectural cladding, and a countless assortment of stamped, rolled, forged, and machined products. With their favorable combination of strength, corrosion resistance, attractive color, and the ability to take a smooth finish, cast brasses are most commonly used in plumbing fixtures and fittings, but they are also widely used for decorative and architectural products, as well as pumps, valves, and propellers and other marine hardware, to name just a few examples. Further information about brasses can be found in the applications areas on http://www.copper.org.

Bronzes, broadly speaking, are copper alloys in which the major alloying element is neither zinc nor nickel. Originally, "bronze" described alloys with tin as the only or principal alloying element. Today, the term is generally used not by itself but with a modifying adjective to describe the main added element. There are four families of wrought bronzes:

- Copper–tin–phosphorus alloys (phosphor bronzes), C50100 through C52400
- Copper–tin–lead–phosphorus alloys (leaded phosphor bronzes), C53200 through C55284
- Copper–aluminum alloys (aluminum bronzes), C60600 through C64400
- Copper–silicon alloys (silicon bronzes), C64700 through C66100

There are also four families of cast bronzes:

- Copper–tin alloys (tin bronzes), C90200 through C91700
- Copper–tin–lead alloys (leaded and high-leaded tin bronzes), C92200 through C92900 and C93100 through C94500, respectively

- Copper–tin–nickel alloys (nickel–tin bronzes), C94700 through C94900
- Copper–aluminum–iron and copper–aluminum–iron–nickel alloys (aluminum bronzes), C95200 through C95810

Wrought bronzes, in sheet and strip form, are used for electrical springs and connectors. Leaded versions are used for a broad variety of machined components requiring high strength and good corrosion resistance. Wrought silicon and aluminum bronzes, for example, are used for bolts, shafts, and other industrial products. Cast bronzes are used for pump, valve and fitting components, wear rings, gears, and products for the chemical, mining, petrochemical, and electroplating industries. Many cast bronzes are used in sleeve bearings, which are discussed later in this chapter. Because of their good castability and favorable weathering characteristics, cast bronzes are the metals most often used in statuary and plaques. Several useful articles dealing with bronzes can be found at http://www.copper.org/industrial/homepage.htm.

Copper–Nickels are copper alloys in which nickel is the principal alloying element. Iron, manganese, niobium (columbium), and other elements may also be present. Both wrought (C70100 through C72900) and cast (C96200 through C96800) copper–nickels exhibit outstanding corrosion and stress–corrosion cracking resistance, especially in seawater. Biofouling resistance (the ability of an alloy to inhibit the attachment of marine organisms) ranges from moderate to excellent, generally increasing with copper content. Copper–nickels are used for condenser and heat exchanger tubes, seawater piping systems, pumps, valves, and fittings. Copper–nickel sheet, applied as cladding or sheathing to seagoing vessels and offshore platform legs, significantly reduces drag caused by the buildup of algae and other marine life. A number of informative studies describing the properties, uses, and fabrication of copper–nickels are available at http://marine.copper.org/.

Nickel–silvers are copper–nickel–zinc alloys or nickel brasses. Nickel gives them the color for which they are named. They offer the good corrosion resistance and favorable mechanical properties inherent to brasses. Wrought nickel–silvers (C73200 through C79900) are available in sheet and strip as well as rod and bar forms. Some alloys produced as rod contain lead for improved machinability. Uses include electrical and electronic connectors, decorative hardware, jewelry, tableware, eyeglass frames, and musical instrument components. Cast nickel–silvers (C97300 through C97800) have long been used in valves and fittings for food and beverage handling equipment.

Leaded coppers are metal–matrix composites (MMCs) rather than true alloys in that lead is not miscible in copper to any appreciable extent. Available only in cast versions (C98200 through C98640), these materials are used exclusively for special-duty bearings.

Finally, there are a number of *miscellaneous copper–zinc alloys* in wrought forms and *special alloys* for cast products whose compositions preclude their inclusion among other alloy families. Wrought alloys (C66400 through C66900) include alloys supplied as sheet and strip, wire, tubular products, and extruded rod and bar for applications such as springs, switch components, condenser tube, valve stems, gears, wear plates, piston rings, propeller shafts, and pole-line hardware. Cast special alloys (C99300 through C99750) include materials designed to provide such unconventional physical or mechanical properties as high damping capacity, ferromagnetic permeability and shape-memory effects.

4 PRODUCT FORMS

Copper and copper alloys are produced in all product forms, although some may be available in several forms while others are only produced in one or two. Basic compositions are often modified to make alloys amenable to manufacture in a particular form, a practice that is sometimes reflected in the final two digits of the UNS numbering codes.

5 ELECTRICAL AND ELECTRONIC WIRE PRODUCTS

Copper's largest single use, exceeding 60% of consumption in many countries, is for electrical applications, and wire and cable products make up the bulk of this market. In the United States, about 45% of all refined copper comes to market as wire and cable, the remaining "electrical" copper being used in other-than-wire products such as busbars, switchgear, pole-line hardware, and tubular windings in large gas-cooled electric generators.

There is little materials selection involved in electrical wire, since virtually all of it is made from electrolytic tough pitch (ETP) copper, C11000. It contains a minimum of 99.90% Cu, making it one of the purest metals in commercial use. Purity largely determines copper's electrical and thermal conductivity, and even minute quantities of most impurity elements severely degrade these properties.

Copper wire products are identified by their end uses. *Building wire*, which includes both insulated and uninsulated wire within buildings and structures, accounts for the largest fraction of copper wire used; *magnet wire* is an insulated or enameled product used in electric motors, transformers, relays, and similar products; *telecommunications cable* includes both voice and data cable; *power cable* is that used by utilities and for heavy industrial conductors; while *automotive wire* is used in wiring harnesses and other purposes. There are also several miscellaneous wire types including apparatus wire, cord sets, electronic wire and cable [local area network (LAN) cable], control and signal wire and cable, and bare wire, among others. Organizations such as the National Electrical Manufacturers Association (NEMA), the Institute of Electrical and Electronic Engineers (IEEE), the National Fire Protection Association (NFPA), and others generally establish specifications for the production and use of electrical wire products.

The conductivity of copper is expressed in units of a percentage of the International Annealed Copper Standard (IACS). This standard is based on an annealed copper wire having a density of 8.89 $\mathrm{g/cm^3}$, 1 m long, weighing 1 g, with a resistance of 0.15328 Ω. This standard was assigned the value 100 at 20°C (68°F). It is not uncommon for commercially pure copper products to have IACS conductivity values greater than 100% because of improved processing techniques that have been developed since the adoption of the standard in 1913.

For example, C11000 exhibits a conductivity 101% IACS. The so-called *oxygen-free* or *OF coppers*, notably C10100 (oxygen-free, electronic) and C10200—there are others—offer slightly higher conductivity, but this is rarely required. Instead, OF coppers are selected for their combination of high conductivity and good weldability. Dissolved oxygen found in ETP and some other coppers causes welding problems. The oxygen contents of OF coppers are low enough to avoid such problems. The addition of deoxidizers such as phosphorus and boron also helps by tying up oxygen in harmless compounds, but deoxidizers—especially phosphorus—also reduce conductivity. Table 3 contains conductivity data, as well as other physical properties, for coppers and selected copper alloys. A listing of physical and mechanical properties for all current UNS copper metals is available on line at http://properties.copper.org/servlet/com.copper.servlet.CDAPropertiesSelectionServlet.

Coppers containing traces of tellurium, cadmium, silver, chromium, zirconium, and titanium, alone or in combination, offer slightly higher mechanical properties. Purity is high enough to classify the metals as coppers rather than high copper alloys. This is reflected in their conductivities, which are typically higher than 90% IACS. Tellurium-bearing copper (C14500) and zirconium copper (C15000) are examples of such coppers. Understanding their uses requires a quick explanation of strengthening in copper.

Coppers are strengthened almost exclusively by cold working, as in wire drawing. The effects of cold work are removed by annealing, i.e., heating the copper above a particular temperature, either intentionally or as a result of high current flow in service. Some secondary elements raise the temperature at which copper begins to soften or anneal. Softening resistance can be important in, for example, pin-type electrical connectors, which are made from these coppers, as well as from high-copper alloys, brasses, bronzes, and other alloys.

A large body of technical data and literature about copper's electrical and electronic applications can be found in the Electrical Energy Efficiency, Power Quality and Telecommunications areas of *The Copper Page,* http://www.copper.org.

6 SHEET, STRIP, AND PLATE PRODUCTS

In contrast to wire products, which are made from a relatively small number of coppers, products produced as sheet, strip, and plate are available in a large selection of coppers and (especially) copper alloys. The uses to which these metals are put can broadly be grouped into architectural and decorative applications, electrical/electronic technologies, and industrial products.

6.1 Architecture

Copper has a long history of use for roofing, flashing and gutters, and other architectural products. In the United States, such products were until recently found mainly on public buildings. Today, use is growing in domestic housing, and copper metals are being selected for new applications such as wall and column cladding.

One reason for copper's growth is that the patinas that form naturally on exposed surfaces over time can now be applied during manufacture, thus providing the architect/designer with an even larger palette of colors and finishes. Information about copper's architectural uses can be accessed at http://architecture.copper.org/.

Table 3 Physical Properties of Selected Coppers and Copper Alloys (Copper Development Association, Inc.)

UNS Alloy	Liquidus, °F (°C)	Solidus, °F (°C)	Density, lb/in³ 68 °F (g/cm³, 20°C)	Specific Gravity	Electrical Resistivity, Ω·cmil/ft, 68°F (Ω-cm, 20°C)	Electrical Conductivity*, % IACS, 68°F (MS/cm, 20°C)	Thermal Conductivity Btu/ft²/ft/h/°F, 68°F (W/m·K, 20°C)	Thermal Expansion Coefficient 10⁻⁶/°F, (10⁻⁶/°C) 68–212°F (20–100°C)	68–392°F (20–200°C)	68–572°F (20–300°C)	Heat Capacity, Btu/lb/°F (J/kg·K)	Elastic Modulus, Tension ksi (MPa)	Modulus of Rigidity ksi (MPa)
C10100	1,981	1,981	0.323	8.94	10.3	101	226.0	9.4	9.6	9.8	0.092	17,000	6,400
C11000 C10910	(1,083)	(1,083)	(8.94)	(8.94)	(1.71)	(0.591)	(391.1)	(16.9)	(17.3)	(17.6)	(393.5)	(117,000)	(44,126)
C10200	1,981	1,981	0.323	8.94	10.6	101	223.0	9.4	9.6	9.78	0.092	17,000	6,400
	(1,083)	(1,083)	(8.94)	(8.94)	(1.76)	(0.591)	(386.0)	(16.9)	(17.3)	(17.6)	(385.15)	(117,000)	(44,126)
C12000	1,981	1,981	0.323	8.94	10.6	98	223.0	9.4	9.6	9.8	0.092	17,000	6,400
C12200	(1,083)	(1,083)	(8.94)	(8.94)	(1.76)	(0.573)	(386.0)	(16.9)	(17.3)	(17.6)	(393.5)	(117,000)	(44,126)
C12510	1,981	1,949	0.323	8.94	11.3	92	202.0	9.3	9.6	9.8	0.092	17,400	6,400
	(1,083)	(1,065)	(8.94)	(8.94)	(1.87)	(0.533)	(346.9)	(16.8)	(17.3)	(17.6)	(393.5)	(119,960)	(44,124)
C14500	1,976	1,924	0.323	8.94	11.2	93	205.0	9.5	9.7	9.9	0.092	17,000	6,400
	(1,080)	(1,051)	(8.94)	(8.94)	(1.86)	(0.544)	(354.8)	(17.1)	(17.5)	(17.8)	(385.15)	(117,000)	(44,126)
C14530	1,976	1,926	0.0323	8.94	10.8	96	225.0	9.4	9.6	9.8	0.092	17,000	6,400
	(1,080)	(1,052)	(8.094)	(8.94)	(1.87)	(0.557)	(328.3)	(16.9)	(17.3)	(17.6)	(385.15)	(117,000)	(44,126)
C15000	1,976	1,796	0.321	8.89	11.2	93	212.0	9.4	—	9.8	0.092	18,700	6,400
	(1,080)	(980)	(8.89)	(8.89)	(1.86)	(0.544)	(366.9)	(16.9)	(—)	(17.6)	(385.15)	(129,000)	(44,126)
C16200	1,969	1,886	0.312	8.89	11.5	90	208.0	9.4	9.6	9.8	0.09	17,000	6,400
	(1,076)	(1,030)	(8.89)	(8.89)	(1.91)	(0.527)	(630)	(1,639)	(17.3)	(17.6)	(376.78)	(117,000)	(44,126)
C17200	1,800	1,590	0.298	8.25	46.2	22	62	—	—	9.9	0.1	18,500	7,300
	(982)	(866)	(8.25)	(8.25)	(7.68)	(0.129)	(107.3)	(—)		(17.8)	(418.64)	(128,000)	(50,330)
17410	1,950	1,875	0.318	8.81	36.9	45	120.0	—	—	—	—	19,000	—
	(1,066)	(1,024)	(8.81)	(8.81)	(6.13)	(0.263)	(207.7)	(—)	(—)	(—)	(—)	(131,000)	(—)
17450	1,960	1,885	0.323	8.94	18.9	45	128.0	—	—	—	—	20,000	—
	(1,075)	(1,032)	(8.94)	(8.94)	(3.12)	(0.263)	(222.0)	(—)	(—)	(—)	(—)	(137,900)	(—)
17460	1,975	1,880	0.318	8.81	20.7	45	128.0	—	—	—	—	20,000	—
	(1,080)	(1,030)	(8.81)	(8.81)	(3.42)	(0.263)	(222.0)	(—)	(—)	(—)	(—)	(137,900)	(—)
C17510	1,955	1,885	0.317	8.77	22.8	45 (48 T)	120.0	—	9.8	—	0.1	20,000	7,500
	(1,068)	(1,029)	(8.77)	(8.77)	(3.79)	[0.263 (0.281 HT)]	(207.7)	(—)	(17.6)	(—)	(418.64)	(137,900)	(51,700)
C18100	1,976	—	0.319	8.83	13.0	80	187.0	9.3	10.2	10.7	0.094	18,200	6,800
	(1,075)	(—)	(8.83)	(8.83)	(2.16)	(0.468)	(323.6)	(16.7)	(18.4)	(19.3)	(393.53)	(125,000)	(46,900)
18200	1,967	1,958	0.321	8.89	13.0	80	187.0	9.8	—	—	0.092	17,000	7,200
18400	(1,075)	(1,070)	(8.89)	(8.89)	(2.16)	(0.468)	(323.6)	(17.6)	(—)	(—)	(385.15)	(117,100)	(49,600)

19210	1,980	—	0.323	8.94	—	80	—	—	—	9.4	0.092	18,200	—
	(1,082)	(—)	(8.94)	(8.94)	(—)	(0.468)	(—)	(—)	(—)	(16.9)	(385.15)	(125,000)	(—)
19400	1,990	1,980	0.322	8.91	15.0	65	150.0	—	—	9.8	0.092	17,500	6,600
	(1,088)	(1,082)	(8.91)	(8.91)	(2.49)	(0.38)	(259.6)			(17.6)	(385.15)	(121,000)	(45,500)
19800	1,963	1,920	0.322	8.91	2.83	61	150.0	—	—	10.2	0.094	11,800	—
	(1,073)	(1,049)	(8.91)	(8.91)	(0.467)	(0.384)	(259.6)	(—)	(—)	(18.4)	(393.0)	(81,300)	(—)
21000	1,950	1,920	0.32	8.86	18.5	56	135.0	—	—	10.0	0.09	17,000	6,400
	(1,066)	(1,049)	(8.86)	(8.86)	(3.08)	(0.328)	(233.6)	(—)	(—)	(18.0)	(376.78)	(117,000)	(44,126)
C22600	1,895	1,840	0.317	8.77	25.9	40	100.0	—	—	10.3	0.09	17,000	6,400
	(1,035)	(1,004)	(8.77)	(8.77)	(4.13)	(0.234)	(173.1)	(—)	(—)	(18.5)	(376.78)	(117,000)	(44,126)
C23000	1,880	1,810	0.316	8.75	28.0	37	92.0	—	—	10.4	0.09	17,000	6,400
	(1,027)	(988)	(8.75)	(8.75)	(4.65)	(0.216)	(159.2)	(—)	(—)	(18.7)	(376.78)	(117,000)	(44,126)
23100													
C26000	1,750	1,680	0.308	8.53	37.0	28	70.0	—	—	11.1	0.09	16,000	6,000
C26130	(954)	(916)	(8.53)	(8.53)	(6.15)	(0.164)	(121.2)	(—)	(—)	(20.0)	(376.78)	(110,000)	(41,426)
C27000	1,710	1,660	0.306	8.47	38.4	27	67.0	—	—	11.3	0.09	15,000	5,600
	(932)	(904)	(8.47)	(8.47)	(6.38)	(0.158)	(116.0)	(—)	(—)	(20.3)	(376.78)	(103,400)	(38,600)
C28000	1,660	1,650	0.303	8.39	37.0	28	71.0	—	—	11.6	0.09	15,000	5,600
	(904)	(899)	(8.39)	(8.39)	(6.15)	(0.164)	(122.9)	(—)	(—)	(20.9)	(376.78)	(103,400)	(38,600)
C34500	1,670	1,630	0.306	8.47	39.9	26	67.0	—	—	11.3	0.09	15,000	5,600
C35300	(910)	(888)	(8.47)	(8.47)	(6.63)	(0.152)	(116.0)	(—)	(—)	(20.3)	(376.78)	(103,400)	(38,600)
C36000	1,650	1,630	0.307	8.5	39.9	26	67.0	—	—	11.4	0.09	14,000	5,300
	(899)	(888)	(8.5)	(8.5)	(6.63)	(0.152)	(116.0)	(—)	(—)	(20.5)	(376.78)	(96,500)	(36,500)
C37700	1,640	1,620	0.305	8.44	38.4	27	69.0	—	—	11.5	0.09	15,000	5,600
	(893)	(882)	(8.44)	(8.44)	(6.38)	(0.158)	(119.4)	(—)	(—)	(20.7)	(376.78)	(103,400)	(38,600)
C46400	1,650	1,630	0.304	8.41	39.9	26	67.0	—	—	11.8	0.09	15,000	5,600
	(899)	(888)	(8.41)	(8.41)	(6.63)	(0.152)	(116.0)	(—)	(—)	(21.2)	(376.78)	(103,400)	(38,600)
C48200	1,650	1,630	0.305	8.44	39.9	26	67.0	—	—	11.8	0.09	15,000	5,600
	(899)	(888)	(8.44)	(8.44)	(6.63)	(0.152)	(116.0)	(—)	(—)	(21.2)	(376.78)	(103,400)	(38,600)
C50725	1,978	1,904	0.322	8.91	—	33	87.0	—	—	9.7	0.09	16,400	6,400
	(1,081)	(1,040)	(8.91)	(8.91)	(—)	(0.191)	(150.7)	(—)	(—)	(17.5)	(376.78)	(113,100)	(44,126)

* As annealed unless otherwise noted.

Table 3 (*Continued*)

UNS Alloy	Liquidus, °F (°C)	Solidus, °F (°C)	Density, lb/in^3 68 °F (g/cm^3, 20°C)	Specific Gravity	Electrical Resistivity, Ω·cmil/ft, 68°F (Ω-cm, 20°C)	Electrical Conductivity*, % IACS, 68°F (MS/cm, 20°C)	Thermal Conductivity Btu/ft^2/ft/h/°F, 68°F (W/m·K, 20°C)	Thermal Expansion Coefficient 10^{-6}/°F, (10^{-6}/°C)			Heat Capacity, Btu/lb/°F (J/kg·K)	Elastic Modulus, Tension ksi (MPa)	Modulus of Rigidity ksi (MPa)
								68–212°F (20–100°C)	68–392°F (20–200°C)	68–572°F (20–300°C)			
C51000	1,920 (1,049)	1,750 (954)	0.32 (8.86)	8.86 (8.86)	69.1 (11.49)	15 (0.088)	40.0 (69.2)	— (—)	— (—)	9.9 (17.8)	0.09 (376.78)	16,000 (110,000)	6,000 (41,400)
C51100	1,945 (1,063)	1,785 (974)	0.32 (8.86)	8.86 (8.86)	52.0 (8.64)	20 (0.117)	48.4 (83.8)	— (—)	— (—)	9.9 (17.8)	0.09 (376.78)	16,000 (110,000)	6,000 (41,400)
C51900	1,900 (1,040)	1,700 (930)	0.319 (8.84)	8.84 (8.84)	74.0 (12.3)	14 (0.081)	38.0 (65.8)	10.0 (18.0)	— (—)	— (—)	0.09 (376.78)	16,000 (110,000)	6,000 (41,400)
C52100	1,880 (1,027)	1,620 (882)	0.318 (8.8)	8.8 (8.8)	79.8 (13.27)	13 (0.076)	36.0 (62.3)	10.1 (18.2)	— (—)	— (—)	0.09 (376.78)	16,000 (110,000)	6,000 (41,400)
C61300	1,915 (1,046)	1,905 (1,041)	0.287 (7.95)	7.95 (7.95)	86.8 (14.43)	12 (0.07)	32.0 (55.4)	— (—)	— (—)	9.0 (16.2)	0.09 (376.78)	17,000 (117,000)	6,400 (44,126)
C61800	1,913 (1,045)	1,904 (1,040)	0.272 (7.53)	7.53 (7.53)	79.8 (13.27)	37.0 (64.0)	13 (0.076)	— (—)	— (—)	9.0 (16.2)	0.09 (376.78)	17,000 (117,000)	6,400 (44,126)
C63000	1,930 (1,054)	1,895 (1,035)	0.274 (7.58)	7.58 (7.58)	116.0 (19.28)	7 (0.041)	22.6 (39.1)	— (—)	— (—)	9.0 (16.2)	0.09 (376.78)	17,500 (121,000)	6,400 (44,126)
C65500	1,880 (1,027)	1,780 (971)	0.308 (8.53)	8.53 (8.53)	148.0 (24.6)	7 (0.041)	21.0 (36.3)	— (—)	— (—)	10.0 (18.0)	0.09 (376.78)	15,000 (103,400)	5,600 (38,600)
C66430	1,880 (1,027)	1,830 (1,000)	0.317 (8.78)	8.78 (8.78)	37.0 (6.16)	28 (0.162)	70.0 (121.0)	10.0 (18.0)	— (—)	— (—)	0.09 (376.78)	16,000 (112,000)	6,000 (42,000)
C67000	1,652 (900)	— (—)	0.302 (8.36)	8.36 (8.36)	47.2 (7.84)	22 (0.128)	57.0 (99.0)	— (—)		11.9 (21.4)	— (—)	— (—)	— (—)
C67500	1,630 (888)	1,590 (866)	0.302 (8.36)	8.36 (8.36)	43.2 (7.18)	24 (0.14)	61.0 (105.6)	— (—)	— (—)	11.8 (21.2)	0.09 (376.78)	15,000 (103,400)	5,600 (38,600)
C70260	1,940 (1,060)	1,905 (1,040)	0.320 (8.86)	8.86 (8.86)	26.0 (42.9)	40 (0.232)	90.0 (155.6)	— (—)	9.0 (16.0)	10.0 (18.0)	0.09 (376.78)	19,000 (131,000)	7,200 (49,600)
C70280	1,967 (1,075)	1,895 (1,035)	0.321 (8.9)	8.9 (8.9)	26.5 (4.31)	40 (0.23)	104.0 (180.0)	— (—)	— (—)	9.7 (17.5)	0.09 (376.0)	19,000 (137,900)	— (—)
C70290	1,958 (1,070)	1,850 (1,010)	0.32 (8.88)	8.88 (8.88)	34.5 (5.74)	30 (0.17)	75.0 (130.0)	— (—)	— (—)	— (—)	0.09 (376.0)	19,000 (137,900)	— (—)
C71000	2,190 (1,199)	2,100 (1,149)	0.323 (8.94)	8.94 (8.94)	160.0 (26.6)	6 (0.038)	21.0 (36.3)	— (—)	— (—)	9.1 (16.4)	0.09 (376.0)	20,000 (138,000)	7,500 (51,700)
C71500	2,260 (1,238)	2,140 (1,171)	0.323 (8.94)	8.94 (8.94)	225.0 (37.4)	4 (0.027)	17.0 (29.4)	— (—)	— (—)	9.0 (13.2)	0.09 (376.0)	21,000 (152,000)	8,300 (57,200)

C72900	2,039 (1,115)	1,742 (950)	0.323 (8.94)	8.94 (8.94)	— (—)	7 (0.046)	17.0 (29.4)	— (—)	— (—)	9.1 (16.4)	0.09 (376.0)	18,500 (128,000)	7,500 (51,700)
C75200	2,030 (1,110)	1,960 (1,071)	0.316 (8.75)	8.75 (8.75)	173.0 (28.76)	6 (0.035)	19.0 (32.9)	— (—)	— (—)	9.0 (16.2)	0.09 (376.0)	18,000 (124,000)	6,800 (46,900)
C76260	1,880 (1,025)	1,800 (980)	0.31 (8.58)	8.58 (8.58)	115.0 (19.2)	9 (0.052)	26.0 (45.0)	— (—)	— (—)	9.0 (16.2)	0.09 (376.0)	18,000 (124,000)	6,800 (46,900)
C80200	1,981	1,948	0.323	8.94	11.3	92	200.0	—	—	9.4	0.09	17,000	—
C81100	(1,083)	(1,064)	(8.94)	(8.94)	(1.87)	(0.538)	(346.1)	(—)	(—)	(16.9)	(376.0)	(117,000)	(—)
C83450	1,860 (1,015)	1,580 (860)	0.319 (8.83)	8.83 (8.83)	— (—)	20 (0.115)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C83600	1,850 (1,010)	1,570 (854)	0.318 (8.83)	8.83 (8.83)	69.1 (11.49)	15 (0.087)	41.6 (72.0)	— (—)	10.0 (18.0)	— (—)	0.09 (376.0)	13,500 (93,100)	— (—)
C84400	1,840 (1,004)	1,549 (843)	0.314 (8.69)	8.69 (8.69)	63.3 (10.53)	16 (0.095)	41.8 (72.4)	— (—)	— (—)	10.0 (18.0)	0.09 (376.0)	13,000 (89,600)	— (—)
C84800	1,750 (954)	1,530 (832)	0.31 (8.58)	8.58 (8.58)	63.1 (10.53)	16 (0.095)	41.6 (72.0)	— (—)	10.0 (18.0)	— (—)	0.09 (376.0)	15,000 (103,400)	— (—)
C85200	1,725 (941)	1,700 (927)	0.307 (8.5)	8.5 (8.5)	57.8 (9.62)	18 (0.104)	48.5 (83.9)	11.5 (20.7)	— (—)	— (—)	0.09 (376.0)	11,000 (75,800)	— (—)
C85400	1,725 (941)	1,700 (927)	0.305 (8.44)	8.44 (8.44)	53.2 (8.85)	20 (0.113)	50.8 (87.9)	11.1 (20.2)	— (—)	— (—)	0.09 (376.0)	12,000 (82,700)	— (—)
C85700	1,725 (941)	1,675 (913)	0.304 (8.41)	8.41 (8.41)	47.0 (7.81)	22 (0.128)	48.5 (83.9)	— (—)	— (—)	12.0 (21.6)	0.09 (376.0)	14,000 (87,000)	— (—)
C86200	1,725 (941)	1,650 (899)	0.288 (7.97)	7.97 (7.97)	136.7 (22.73)	8 (0.044)	20.5 (35.5)	— (—)	— (—)	12.0 (21.6)	0.09 (376.0)	15,000 (103,400)	— (—)
C86300	1,693 (923)	1,625 (885)	0.283 (7.83)	7.83 (7.83)	130.8 (21.74)	8 (0.046)	20.5 (35.5)	— (—)	— (—)	12.0 (21.6)	0.09 (376.0)	14,200 (97,900)	— (—)
C86400	1,616 (880)	1,583 (862)	0.301 (8.33)	8.33 (8.33)	54.2 (9.01)	19 (0.111)	51.0 (88.3)	— (—)	11.0 (19.8)	— (—)	0.09 (376.0)	14,200 (96,500)	— (—)
C87300	1,780 (971)	1,580 (860)	0.302 (8.36)	8.36 (8.36)	171.9 (285.7)	6 (0.035)	16.4 (28.4)	— (—)	— (—)	10.9 (19.6)	0.09 (376.0)	15,000 (103,000)	— (—)

* As annealed unless otherwise noted.

Table 3 (*Continued*)

UNS Alloy	Liquidus, °F (°C)	Solidus, °F (°C)	Density, lb/in^3 68 °F (g/cm^3, 20°C)	Specific Gravity	Electrical Resistivity, Ω·cmil/ft, 68°F (Ω-cm, 20°C)	Electrical Conductivity*, % IACS, 68°F (MS/cm, 20°C)	Thermal Conductivity Btu/ft^2/ft/h/°F, 68°F (W/m·K, 20°C)	Thermal Expansion Coefficient 10^{-6}/°F, (10^{-6}/°C)			Heat Capacity, Btu/lb/°F (J/kg·K)	Elastic Modulus, Tension ksi (MPa)	Modulus of Rigidity ksi (MPa)
								68–212°F (20–100°C)	68–392°F (20–200°C)	68–572°F (20–300°C)			
C87500	1,680 (916)	1,510 (821)	0.299 (8.28)	8.28 (8.28)	154.2 (25.64)	6 (0.039)	16.0 (27.7)	— (—)	— (—)	10.9 (19.6)	0.09 (376.0)	15,400 (106,000)	— (—)
C89510	1,871 (1,021)	371 (206)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C90300	1,832 (1,000)	1,570 (854)	0.318 (8.8)	8.8 (8.8)	87.2 (14.49)	12 (0.069)	43.2 (74.8)	— (—)	10.0 (18.0)	— (—)	0.09 (376.0)	14,000 (96,500)	— (—)
C92200	1,810 (988)	1,518 (826)	0.312 (8.64)	8.64 (8.64)	72.5 (12.0)	14 (0.083)	40.2 (69.6)	— (—)	— (—)	10.0 (18.0)	0.09 (377.0)	14,000 (96,500)	— (—)
C92300	1,830 (999)	1,570 (854)	0.317 (8.77)	8.77 (8.77)	85.9 (14.29)	12 (0.07)	43.2 (74.8)	— (—)	10.0 (18.0)	— (—)	0.09 (377.0)	14,000 (96,500)	— (—)
C92600	1,800 (982)	1,550 (843)	0.315 (8.72)	8.72 (8.72)	115.7 (19.23)	9 (0.052)	— (—)	— (—)	10.0 (18.0)	— (—)	0.09 (377.0)	15,000 (103,400)	— (—)
C93200	1,790 (977)	1,570 (854)	0.322 (8.91)	8.91 (8.91)	85.9 (14.29)	12 (0.07)	33.6 (58.2)	10.0 (18.0)	— (—)	— (—)	0.09 (377.0)	14,500 (100,000)	— (—)
C93500	1,830 (999)	1,570 (854)	0.32 (8.86)	8.86 (8.86)	68.4 (11.36)	15 (0.088)	40.7 (70.4)	— (—)	9.9 (17.8)	— (—)	0.09 (377.0)	14,500 (100,000)	— (—)
C93700	1,705 (929)	1,403 (762)	0.32 (8.86)	8.86 (8.86)	102.0 (16.95)	10 (0.059)	27.1 (46.9)	— (—)	10.3 (18.5)	— (—)	0.09 (377.0)	11,000 (75,800)	— (—)
C93800	1,730 (943)	1,570 (854)	0.334 (9.25)	9.25 (9.25)	91.1 (15.15)	11 (0.066)	30.2 (52.3)	— (—)	10.3 (18.5)	— (—)	0.09 (377.0)	10,500 (72,400)	— (—)
C94300	— (—)	— (—)	0.336 (9.3)	9.3 (9.3)	113.5 (18.87)	9 (0.053)	36.2 (62.7)	— (—)	— (—)	— (—)	0.09 (377.0)	10,500 (72,400)	— (—)
C95200	1,913 (1,045)	1,907 (1,042)	0.276 (7.64)	7.64 (7.64)	94.0 (15.63)	11 (0.064)	29.1 (50.4)	— (—)	— (—)	9.0 (16.2)	0.09 (377.0)	15,000 (103,400)	— (—)
C95300	1,913 (1,045)	1,904 (1,040)	0.272 (7.53)	7.53 (7.53)	80.2 (13.33)	13 (0.075)	36.3 (62.8)	— (—)	— (—)	9.0 (16.2)	0.09 (377.0)	16,000 (110,000)	— (—)
C95400	1,900 (1,038)	1,880 (1,027)	0.269 (7.45)	7.45 (7.45)	80.2 (13.33)	13 (0.075)	33.9 (58.7)	— (—)	— (—)	9.0 (16.2)	0.09 (377.0)	15,500 (107,000)	— (—)
C95500	1,930 (1,054)	1,900 (1,038)	0.272 (7.53)	7.53 (7.53)	122.8 (20.41)	8 (0.049)	24.2 (41.9)	— (—)	— (—)	9.0 (16.2)	0.09 (377.0)	16,000 (110,000)	— (—)

C95700	1,814	1,742	0.272	7.53	334.2	3	7.0	—	—	9.8	0.105	18,000	—
	(990)	(950)	(7.53)	(7.53)	(55.56)	(0.018)	(12.1)	(—)	(—)	(17.6)	(440.0)	(124,000)	(—)
C95800	1,940	1,910	0.276	7.64	146.7	7	20.8	—	—	9.0	0.105	16,500	—
	(1,060)	(1,043)	(7.64)	(7.64)	(24.39)	(0.041)	(36.0)	(—)	(—)	(16.2)	(440.0)	(114,000)	(—)
C96400	2,260	2,140	0.323	8.94	214.8	5	16.4	—	—	9.0	0.09	21,000	—
	(1,238)	(1,171)	(8.94)	(8.94)	(35.71)	(0.028)	(28.5)	(—)	(—)	(16.2)	(377.0)	(145,000)	(—)
C97300	1,904	1,850	0.321	8.89	182.3	6	16.5	—	—	9.0	0.09	16,000	—
	(1,040)	(1,010)	(8.89)	(8.89)	(30.3)	(0.033)	(28.6)	(—)	(—)	(16.2)	(377.0)	(110,000)	(—)
C97400	2,012	1,958	0.32	8.86	188.0	6	15.8	—	—	9.2	0.09	16,000	—
	(1,100)	(1,070)	(8.86)	(8.86)	(31.25)	(0.033)	(27.3)	(—)	(—)	(16.6)	(377.0)	(110,000)	(—)
C97600	2,089	2,027	0.321	8.89	207.4	5	13.0	—	—	9.3	0.09	16,000	—
	(1,143)	(1,108)	(8.89)	(8.89)	(34.48)	(0.029)	(22.6)	(—)	(—)	(16.7)	(377.0)	(131,000)	(—)
C97800	2,156	2,084	0.32	8.86	231.4	4	14.7	—	—	9.7	0.09	19,000	—
	(1,180)	(1,140)	(8.86)	(8.86)	(38.46)	(0.026)	(25.4)	(—)	(—)	(17.5)	(377.0)	(131,000)	(—)

* As annealed unless otherwise noted.

6.2 Electrical and Electronic Alloys

Electrical and electronic connectors, contacts, leadframes and components of switches, relays, and similar products form another important use of copper metals. The principal property of concern here is electrical conductivity, followed by formability, corrosion resistance, and spring properties, i.e., the ability to maintain required contact forces. (Thermal conductivity roughly parallels electrical conductivity, and like electrical conductivity, it decreases with increasing concentrations of alloying elements.) The ability to retain mechanical properties at moderately elevated temperatures is also important. This property, known as stress-relaxation resistance, is important in high-current-carrying components, components that require constant contact forces over time and products that are subjected to high "burn-in" currents during testing. Mechanical properties of sheet, strip, and plate alloys, including those used in electrical and electronic applications, are listed in Table 4.

The copper alloy most commonly used in electrical connectors is cartridge brass, UNS C26000. The alloy's electrical conductivity is only 28% that of pure copper, but that is sufficient for many applications. C26000 is also the most formable of the brasses, and its ductility enables complex connectors to be produced at high speed. Brasses containing less than the 30% zinc found in C26000 have higher conductivity, but they are somewhat weaker and less formable. No practical benefit is gained by adding more than 30% zinc to brass for electrical connectors.

Phosphor bronzes comprise the next-most popular group of connector alloys. The phosphor bronzes trade-off conductivity for higher strength and are used to provide high contact forces when electrical conductivity is not a prime concern. Conversely, they can provide equivalent contact force with less metal. With innovative design, reducing the weight of material required for each contact can often offset the cost penalty for alloys carrying a higher price per pound.

Unlike the brasses, which offer a wide range of electrical conductivity (28–56% IACS), conductivities of phosphor bronzes range from only 11 up to 20% IACS, although there are exceptions. Conductivity of phosphor bronze A (5% tin, UNS C51000), the leading alloy, is 15% IACS, only about one-half that of C26000, but it is twice as strong.

Phosphor bronze C (C52100) is another popular alloy in this family. With 8% tin and a small amount of phosphorus, it is stronger but not so highly conducting as C51000. Alloy UNS C51100 offers somewhat higher conductivity than C51000 and C52100 and is also frequently used. These alloys are available in strip form in a variety of tempers. See the data tables for conductivity and mechanical properties.

Coppers containing small quantities of tellurium, zirconium, magnesium, cadmium (now in disfavor due to its adverse effects on the environment), chromium, and iron, in some cases in combination with phosphorus, can provide conductivity of around 80–90% IACS with yield strengths between about 35 and 70 ksi (240 and 480 MPa).

High-copper alloys offer combinations of high conductivity and high mechanical properties. Typical alloys in this class include chromium–coppers such as C18200 and C18400; copper–iron–phosphorus alloys (C19210, C19400, and

Table 4 Mechanical Properties of Selected Sheet and Strip Alloys (Copper Development Association, Inc.)

UNS Alloy	Temper	Section Size, in. (mm)	Cold Work (%)	Typ/ Min	Temp °F (°C)	Tensile Strength ksi (MPa)	Yield Strength (0.5% ext. under load) ksi (MPa)	Yield Strength (0.2% offset) ksi (MPa)	El %	Rockwell Hardness B	C	F	30T	Vickens Hard. 500 kg	Shear Strength ksi (MPa)	Fatigue Strength* ksi (MPa)
C10100	H00	0.04	0	TYP	68	36	28	—	30	10	—	60	25	—	25	—
		(1.0)			(20)	(248)	(193)	(—)	(30)	(10)	(—)	(60)	(25)	(—)	(172)	(—)
	H01	0.04	0	TYP	68	38	30	—	25	25	—	70	36	—	25	—
		(1.0)			(20)	(262)	(207)	(—)	(25)	(25)	(—)	(70)	(36)	(—)	(172)	(—)
	H04	0.04	0	TYP	68	50	45	—	6	50	—	90	57	—	28	13
		(1.0)			(20)	(345)	(310)	(—)	(6)	(50)	(—)	(90)	(57)	(—)	(193)	(90)
	H08	0.04	0	TYP	68	55	50	—	4	60	—	94	63	—	29	14
		(1.0)			(20)	(379)	(345)	(—)	(4)	(60)	(—)	(94)	(63)	(—)	(200)	(97)
	M20	0.04	0	TYP	68	34	10	—	45	—	—	45	—	—	23	—
		(1.0)			(20)	(234)	(69)	(—)	(45)	(—)	(—)	(45)	(—)	(—)	(159)	(—)
	OS025	0.04	0	TYP	68	34	11	—	45	—	—	45	—	—	23	11
		(1.0)			(20)	(234)	(76)	(—)	(45)	(—)	(—)	(45)	(—)	(—)	(159)	(76)
	OS050	0.04	0	TYP	68	32	10	—	45	—	—	40	—	—	22	—
		(1.0)			(20)	(221)	(69)	(—)	(45)	(—)	(—)	(40)	(—)	(—)	(152)	(—)
C10910	H00	0.04	0	TYP	68	36	28	—	30	10	—	60	25	—	—	—
		(1.0)			(20)	(248)	(193)	(—)	(30)	(10)	(—)	(60)	(25)	(—)	(—)	(—)
	H01	0.04	0	TYP	68	38	30	—	25	25	—	70	36	—	—	—
		(1.0)			(20)	(262)	(207)	(—)	(25)	(25)	(—)	(70)	(36)	(—)	(—)	(—)
	H02	0.04	0	TYP	68	42	36	—	14	40	—	84	50	—	—	—
		(1.0)			(20)	(290)	(248)	(—)	(14)	(40)	(—)	(84)	(50)	(—)	(—)	(—)
	H04	0.04	0	TYP	68	50	45	—	6	50	—	90	57	—	—	—
		(1.0)			(20)	(345)	(310)	(—)	(6)	(50)	(—)	(90)	(57)	(—)	(—)	(—)
	H08	0.04	0	TYP	68	55	50	—	4	60	—	94	63	—	—	—
		(1.0)			(20)	(379)	(345)	(—)	(4)	(60)	(—)	(94)	(63)	(—)	(—)	(—)
	H10	0.04	0	TYP	68	57	53	—	4	62	—	95	64	—	—	—
		(1.0)			(20)	(20,393)	(365)	(—)	(4)	(62)	(—)	(95)	(64)	(—)	(—)	(—)
	M20	0.04	0	TYP	68	34	10	—	45	62	—	45	64	—	—	—
		(1.0)			(20)	(234)	(69)	(—)	(45)	(62)	(—)	(45)	(64)	(—)	(—)	(—)
	OS025	0.04	0	TYP	68	34	11	—	45	62	—	45	64	—	—	—
		(1.0)			(20)	(234)	(76)	(—)	(45)	(62)	(—)	(45)	(64)	(—)	(—)	(—)

Table 4 **(*Continued*)**

UNS Alloy	Temper	Section Size, in. (mm)	Cold Work (%)	Typ/ Min	Temp °F (°C)	Tensile Strength ksi (MPa)	Yield Strength (0.5% ext. under load) ksi (MPa)	Yield Strength (0.2% offset) ksi (MPa)	El %	Rockwell Hardness B	C	F	30T	Vickens Hard. 500 kg	Shear Strength ksi (MPa)	Fatigue Strength* ksi (MPa)
	OS050	0.04	0	TYP	68	32	10	—	45	62	—	40	64	—	—	—
		(1.0)			(20)	(221)	(69)	(—)	(45)	(62)	(—)	(40)	(64)	(—)	(—)	(—)
	OS050	0.25	0	TYP	68	32	10	—	50	50	—	40	64	—	—	—
		(6.4)			(20)	(221)	(69)	(—)	(50)	(50)	(—)	(40)	(64)	(—)	(—)	(—)
19010	H01	—	—	MIN	68	52—64	—	40	12	—	—	—	—	100–130	—	—
	R360				(20)	(360–430)	(—)	(275)	(12)	(—)	(—)	(—)	(—)	(100–130)	(—)	(—)
	H02	—	—	MIN	68	60–70	—	54	10	—	—	—	—	125–155	—	—
	R410	—	—		(20)	(410–470)	(—)	(370)	(10)	(—)	(—)	(—)	(—)	(125–155)	(—)	(—)
	H03	—	—	MIN	68	67–77	—	62	8	—	—	—	—	135–165	—	—
	R460	(—)	(—)		(20)	(460–520)	(—)	(410)	(8)	(—)	(—)	(—)	(—)	(135–165)	(—)	(—)
	H04	—	—	MIN	68	71–81	—	66	7	—	—	—	—	145–175	—	—
	R490	(—)	(—)		(20)	(490–560)	(—)	(435)	(7)	(—)	(—)	(—)	(—)	(145–175)	(—)	(—)
	H06	—	—	MIN	68	75–86	—	72	6	—	—	—	—	150–180	—	—
	R520	(—)	(—)		(20)	(520–580)	(—)	(460)	(6)	(—)	(—)	(—)	(—)	(150–180)	(—)	(—)
	H08	—	—	MIN	68	84	—	78	6	—	—	—	—	170–200	—	—
	R580	(—)	(—)		(20)	(580—650)	(—)	(520)	(6)	(—)	(—)	(—)	(—)	(170–200)	(—)	(—)
	TM03	—	—	MIN	68	67–77	—	50	12	—	—	—	—	135–165	—	—
					(20)	(460–520)	(—)	(340)	(12)	(—)	(—)	(—)	(—)	(135–165)	(—)	(—)
	TM04	—	—	MIN	68	71–81	—	60	10	—	—	—	—	145–175	—	—
		(—)	(—)		(20)	(490–560)	(—)	(410)	(10)	(—)	(—)	(—)	(—)	(145–175)	(—)	(—)
	TM06	—	—	MIN	68	75–86	—	64	8	—	—	—	—	150–180	—	—
		(—)	(—)		(20)	(520–580)	(—)	(440)	(8)	(—)	(—)	(—)	(—)	(150–180)	(—)	(—)
	TM08	—	—	MIN	68	84	—	74	6	—	—	—	—	170—200	—	—
		(—)	(—)		(20)	(580)	(—)	(510)	(6)	(—)	(—)	(—)	(—)	(170–200)	(—)	(—)
C12000	H00	0.04	0	TYP	68	36	28	—	30	10	—	60	25	—	25	—
C12200		(1.0)			(20)	(248)	(193)	(—)	(30)	(10)	(—)	(60)	(25)	(—)	(172)	(—)
	H01	0.04	0	TYP	68	38	30	—	25	25	—	70	36	—	25	—
		(1.0)			(20)	(262)	(207)	(—)	(25)	(25)	(—)	(70)	(36)	(—)	(172)	(—)
	H02	0.04	0	TYP	68	42	36	—	14	40	—	84	50	—	26	13
		(1.0)			(20)	(290)	(248)	(—)	(14)	(40)	(—)	(84)	(50)	(—)	(179)	(90)

	H04	0.04 (1.0)	0	TYP	68 (20)	50 (345)	45 (310)	— (—)	6 (6)	50 (50)	— (—)	90 (90)	57 (57)	— (—)	28 (193)	13 (90)
	H08	0.04 (1.0)	0	TYP	68 (20)	55 (379)	50 (345)	— (—)	4 (4)	60 (60)	— (—)	94 (94)	63 (63)	— (—)	29 (200)	14 (97)
	M20	0.04 (1.0)	0	TYP	68 (20)	34 (234)	10 (69)	— (—)	45 (45)	— (—)	— (—)	45 (45)	— (—)	— (—)	23 (159)	— (—)
	OS025	0.04 (1.0)	0	TYP	68 (20)	34 (234)	11 (76)	— (—)	45 (45)	— (—)	— (—)	45 (45)	— (—)	— (—)	23 (159)	11 (76)
	OS050	0.04 (1.0)	0	TYP	68 (20)	32 (221)	10 (69)	— (—)	45 (45)	— (—)	— (—)	40 (40)	— (—)	— (—)	22 (152)	— (—)
125100	H01	0.0030 (0.076)	0	TYP	68 (20)	40 (276)	— (—)	28 (193)	5 (5)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H04	0.0030 (0.076)	0	TYP	68 (20)	51 (352)	— (—)	49 (338)	1 (1)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H08	0.0030 (0.076)	0	TYP	68 (20)	59 (4078)	— (—)	58 (400)	0 (0)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H10	0.0030 (0.076)	0	TYP	68 (20)	59 (407)	— (—)	— (—)	0 (0)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	OS015	0.0030 (0.076)	0	TYP	68 (20)	33 (228)	— (—)	12 (83)	15 (15)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C14530	H01	0.02 (0.51)	0	TYP	68 (20)	40 (276)	— (—)	28 (193)	23 (23)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H02	0.02 (0.51)	0	TYP	68 (20)	45 (310)	— (—)	39 (269)	12 (12)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H03	0.02 (0.51)	0	TYP	68 (20)	48 (331)	— (—)	45 (310)	6 (6)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H04	0.02 (0.51)	0	TYP	68 (20)	51 (352)	— (—)	49 (338)	3 (3)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C14530	H06	0.02 (0.51)	0	TYP	68 (20)	55 (379)	— (—)	53 (365)	2 (2)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)

Table 4 (*Continued*)

UNS Alloy	Temper	Section Size, in. (mm)	Cold Work (%)	Typ/ Min	Temp °F (°C)	Tensile Strength ksi (MPa)	Yield Strength (0.5% ext. under load) ksi (MPa)	Yield Strength (0.2% offset) ksi (MPa)	El %	Rockwell Hardness				Vickens Hard. 500 kg	Shear Strength ksi (MPa)	Fatigue Strength* ksi (MPa)
										B	C	F	30T			
	H08	0.02 (0.51)	0	TYP	68 (20)	59 (407)	— (—)	58 (400)	1 (1)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H10	0.02 (0.51)	0	TYP	68 (20)	— (—)	— (—)	— (—)	1 (1)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	OS015	0.02 (0.51)	0	TYP	68 (20)	33 (228)	— (—)	12 (83)	40 (40)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C17200	TB00	0.0 (0.0)	0	TYP	68 (20)	70 (483)	— (—)	32 (221)	45 (45)	60 (60)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TD01	0.188 (4.78)	0	TYP	68 (20)	80 (552)	— (—)	70 (483)	25 (25)	80 (80)	— (—)	— (—)	70 (70)	— (—)	— (—)	— (—)
	TD04	0.188 (4.78)	0	TYP	68 (20)	110 (758)	— (—)	104 (717)	5 (5)	99 (99)	— (—)	— (—)	81 (81)	— (—)	— (—)	— (—)
	TF00	0.188 (4.78)	0	TYP	68 (20)	175 (1207)	— (—)	155 (1069)	6 (6)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TH01	0.188 (4.78)	0	TYP	68 (20)	185 (1276)	— (—)	165 (1138)	4 (4)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TH04	0.188 (4.78)	0	TYP	68 (20)	200 (1379)	— (—)	180 (1241)	2 (2)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TM00	0.188 (4.78)	0	TYP	68 (20)	105 (724)	— (—)	82 (565)	20 (20)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TM04	0.188 (4.78)	0	TYP	68 (20)	142 (979)	— (—)	122 (841)	12 (12)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TM06	0.188 (4.78)	0	TYP	68 (20)	168 (1158)	— (—)	148 (1020)	7 (7)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TM08	0.188 (4.78)	0	TYP	68 (20)	182 (1255)	— (—)	160 (1103)	6 (6)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
17410	TH02	0.0 (0.0)	0	TYP	68 (20)	105 (724)	— (—)	90 (621)	15 (15)	93 (93)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TH04	0.0 (0.0)	0	TYP	68 (20)	120 (827)	— (—)	110 (758)	12 (12)	102 (102)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)

17450	TH02	0.0	0	TYP	68	100	—	85	15	93	—	—	—	—	—	—
		(0.0)			(20)	(689)	(—)	(586)	(15)	(93)	(—)	(—)	(—)	(—)	(—)	(—)
17460	TH04	0.188	0	TYP	68	130	—	115	12	103	—	—	—	—	—	—
		(4.8)			(20)	(896)	(—)	(793)	(12)	(103)	(—)	(—)	(—)	(—)	(—)	(—)
17510	TB00	0.0	0	TYP	68	45	25	—	28	32	—	—	36	—	—	—
		(0.0)			(20)	(310)	(172)	(—)	(28)	(32)	(—)	(—)	(36)	(—)	(—)	(—)
	TD04	0.0	0	TYP	68	78	70	—	5	83	—	—	72	—	—	—
		0.0			20	538	483	—	5	83	—	—	72	—	—	—
	TF00	0.0	0	TYP	68	110	90	—	12	96	—	—	80	—	—	—
		(0.0)			(20)	(758)	(621)	(—)	(12)	(96)	(—)	(—)	(80)	(—)	(—)	(—)
	TH04	0.0	0	TYP	68	115	110	—	8	98	—	—	81	—	—	—
		(0.0)			(20)	(793)	(758)	(—)	(8)	(98)	(—)	(—)	(81)	(—)	(—)	(—)
C18200	TB00	0.04	0	TYP	68	34	19	—	40	16	—	—	—	—	—	—
C18400		(1.0)			(20)	(234)	(131)	(—)	(40)	(16)	(—)	(—)	(—)	(—)	(—)	(—)
	TD00	0.04	50	TYP	68	53	51	—	6	66	—	—	—	—	—	—
		(1.0)			(20)	(365)	(352)	(—)	(6)	(66)	(—)	(—)	(—)	(—)	(—)	(—)
	TF00	0.04	50	TYP	68	51	36	—	22	59	—	—	—	—	—	—
		(1.0)			(20)	(352)	(248)	(—)	(22)	(59)	(—)	(—)	(—)	(—)	(—)	(—)
	TH01	0.04	50	TYP	68	67	59	—	14	79	—	—	—	—	—	—
		(1.0)			(20)	(462)	(407)	(—)	(14)	(79)	(—)	(—)	(—)	(—)	(—)	(—)
C19210	H01	0.018	0	TYP	68	50	—	48	13	—	—	—	—	—	—	—
		(0.46)			(20)	(343)	(—)	(334)	(13)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	H02	0.018	0	TYP	68	57	—	56	6	—	—	—	—	—	—	—
		(0.46)			(20)	(392)	(—)	(383)	(6)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	H04	0.018	0	TYP	68	64	—	63	3	—	—	—	—	—	—	—
		(0.46)			(20)	(441)	(—)	(432)	(3)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	H08	0.018	0	TYP	68	71	—	70	2	—	—	—	—	—	—	—
		(0.46)			(20)	(490)	(—)	(481)	(2)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	O50	0.018	0	TYP	68	43	—	22	38	—	—	—	—	—	—	—
		(0.46)			(20)	(296)	(—)	(152)	(38)	(—)	(—)	(—)	(—)	(—)	(—)	(—)

Table 4 (*Continued*)

UNS Alloy	Temper	Section Size, in. (mm)	Cold Work (%)	Typ/ Min	Temp °F (°C)	Tensile Strength ksi (MPa)	Yield Strength (0.5% ext. under load) ksi (MPa)	Yield Strength (0.2% offset) ksi (MPa)	El %	Rockwell Hardness				Vickens Hard. 500 kg	Shear Strength ksi (MPa)	Fatigue Strength* ksi (MPa)
										B	C	F	30T			
19400	H02	0.04 (1.0)	0	TYP	68 (20)	60 (414)	50 (345)	53 (365)	9 (9)	68 (68)	— (—)	— (—)	66 (66)	— (—)	— (—)	— (—)
	H04	0.04 (1.0)	0	TYP	68 (20)	67 (462)	— (—)	63 (434)	4 (4)	73 (73)	— (—)	— (—)	69 (69)	— (—)	— (—)	21 (145)
19400	H08	0.04 (1.0)	0	TYP	68 (20)	73 (503)	— (—)	71 (486)	2 (2)	75 (75)	— (—)	— (—)	72 (72)	— (—)	— (—)	22 (148)
	H10	0.04 (1.0)	0	TYP	68 (20)	76 (524)	— (—)	73 (503)	2 (2)	77 (77)	— (—)	— (—)	74 (74)	— (—)	— (—)	21 (141)
	O60	0.025 (0.64)	0	TYP	68 (20)	45 (310)	— (—)	24 (165)	32 (32)	38 (38)	— (—)	— (—)	— (—)	— (—)	— (—)	16 (110)
C19800	H02	0.0 (0.0)	0	TYP	68 (20)	63 (433)	— (—)	61 (423)	12 (12)	— (—)	— (—)	— (—)	— (—)	131 (131)	— (—)	— (—)
	H04	0.012 (0.32)	0	TYP	68 (20)	72 (495)	— (—)	69 (478)	10 (10)	— (—)	— (—)	— (—)	— (—)	149 (149)	— (—)	— (—)
	H06	0.0 (0.0)	0	TYP	68 (20)	80 (554)	— (—)	80 (551)	9 (9)	— (—)	— (—)	— (—)	— (—)	166 (166)	— (—)	— (—)
C21000	H01	0.04 (1.0)	0	TYP	68 (20)	42 (290)	32 (221)	— (—)	25 (25)	38 (38)	— (—)	— (—)	44 (44)	— (—)	32 (221)	— (—)
	H04	0.04 (1.0)	0	TYP	68 (20)	56 (386)	50 (345)	— (—)	5 (5)	64 (64)	— (—)	— (—)	60 (60)	— (—)	37 (255)	— (—)
	H08	0.04 (1.0)	0	TYP	68 (20)	64 (441)	58 (400)	— (—)	4 (4)	73 (73)	— (—)	— (—)	66 (66)	— (—)	40 (276)	— (—)
	OS015	0.04 (1.0)	0	TYP	68 (20)	38 (262)	14 (97)	— (—)	42 (42)	— (—)	— (—)	60 (60)	15 (15)	— (—)	30 (207)	— (—)
	OS035	0.04 (1.0)	0	TYP	68 (20)	35 (241)	11 (76)	— (—)	45 (45)	— (—)	— (—)	52 (52)	4 (4)	— (—)	28 (193)	— (—)
	OS050	0.04 (1.0)	0	TYP	68 (20)	34 (234)	10 (69)	— (—)	45 (45)	— (—)	— (—)	46 (46)	— (—)	— (—)	— (—)	— (—)

C22000	H01	0.04	0	TYP	68	45	35	—	25	42	—	—	44	—	—	—
		(1.0)			(20)	(310)	(241)	(—)	(25)	(42)	(—)	(—)	(44)	(—)	(—)	(—)
	H04	0.04	0	TYP	68	61	54	—	5	70	—	—	63	—	—	—
		(1.0)			(20)	(421)	(372)	(—)	(5)	(70)	(—)	(—)	(63)	(—)	(—)	(—)
	H08	0.04	0	TYP	68	72	62	—	3	78	—	—	69	—	—	—
		(1.0)			(20)	(496)	(427)	(—)	(3)	(78)	(—)	(—)	(69)	(—)	(—)	(—)
	M20	0.04	0	TYP	68	39	14	—	44	—	—	60	—	—	—	—
		(1.0)			(20)	(269)	(97)	(—)	(44)	(—)	(—)	(60)	(—)	(—)	(—)	(—)
	OS015	0.04	0	TYP	68	41	15	—	42	—	—	65	26	—	—	—
		(1.0)			(20)	(283)	(103)	(—)	(42)	(—)	(—)	(65)	(26)	(—)	(—)	(—)
C22600	H01	0.04	0	TYP	68	47	37	—	25	47	—	—	—	—	—	—
		(1.0)			(20)	(324)	(255)	(—)	(25)	(47)	(—)	(—)	(—)	(—)	(—)	(—)
	H04	0.04	0	TYP	68	66	56	—	5	73	—	—	—	—	—	—
		(1.0)			(20)	(455)	(386)	(—)	(5)	(73)	(—)	(—)	(—)	(—)	(—)	(—)
	H08	0.04	0	TYP	68	79	62	—	4	82	—	—	—	—	—	—
		(1.0)			(20)	(545)	(427)	(—)	(4)	(82)	(—)	(—)	(—)	(—)	(—)	(—)
	OS015	0.04	0	TYP	68	44	16	—	42	—	—	68	—	—	—	—
		(1.0)			(20)	(303)	(110)	(—)	(42)	(—)	(—)	(68)	(—)	(—)	(—)	(—)
	OS035	0.04	0	TYP	68	40	13	—	45	—	—	59	—	—	—	—
		(1.0)			(20)	(276)	(90)	(—)	(45)	(—)	(—)	(59)	(—)	(—)	(—)	(—)
	OS050	0.04	0	TYP	68	39	11	—	46	—	—	55	—	—	—	—
		(1.0)			(20)	(269)	(76)	(—)	(46)	(—)	(—)	(55)	(—)	(—)	(—)	(—)
C23000	H02	0.04	0	TYP	68	57	49	—	12	65	—	—	60	—	—	—
		(1.0)			(20)	(393)	(338)	(—)	(12)	(65)	(—)	(—)	(60)	(—)	(—)	(—)
	H04	0.04	0	TYP	68	70	57	—	5	77	—	—	68	—	—	—
		(1.0)			(20)	(483)	(393)	(—)	(5)	(77)	(—)	(—)	(68)	(—)	(—)	(—)
	H08	0.04	0	TYP	68	84	63	—	3	86	—	—	74	—	—	—
		(1.0)			(20)	(579)	(434)	(—)	(3)	(86)	(—)	(—)	(74)	(—)	(—)	(—)
	OS015	0.04	0	TYP	68	45	18	—	42	—	—	71	38	—	—	—
		(1.0)			(20)	(310)	(124)	(—)	(42)	(—)	(—)	(71)	(38)	(—)	(—)	(—)

Table 4 ***(Continued)***

UNS Alloy	Temper	Section Size, in. (mm)	Cold Work (%)	Typ/ Min	Temp °F (°C)	Tensile Strength ksi (MPa)	Yield Strength (0.5% ext. under load) ksi (MPa)	Yield Strength (0.2% offset) ksi (MPa)	El %	Rockwell Hardness B	Rockwell Hardness C	Rockwell Hardness F	Rockwell Hardness 30T	Vickens Hard. 500 kg	Shear Strength ksi (MPa)	Fatigue Strength* ksi (MPa)
	OS035	0.04	0	TYP	68	41	14	—	46	—	—	63	22	—	—	—
		(1.0)			(20)	(283)	(97)	(—)	(46)	(—)	(—)	(63)	(22)	(—)	(—)	(—)
	OS050	0.04	0	TYP	68	40	12	—	47	—	—	59	14	—	—	—
		(1.0)			(20)	(276)	(83)	(—)	(47)	(—)	(—)	(59)	(14)	(—)	(—)	(—)
	OS070	0.04	0	TYP	68	39	10	—	48	—	—	56	10	—	—	—
		(1.0)			(20)	(269)	(69)	(—)	(48)	(—)	(—)	(56)	(10)	(—)	(—)	(—)
C26000	H01	0.04	0	TYP	68	54	40	—	43	55	—	—	54	—	36	—
C26130		(1.0)			(20)	(372)	(276)	(—)	(43)	(55)	(—)	(—)	(54)	(—)	(248)	(—)
	H02	0.04	0	TYP	68	62	52	—	25	70	—	—	65	—	40	18
		(1.0)			(20)	(427)	(359)	(—)	(25)	(70)	(—)	(—)	(65)	(—)	(276)	(124)
	H01	0.04	0	TYP	68	54	40	—	43	55	—	—	54	—	—	—
		(1.0)			(20)	(372)	(276)	(—)	(43)	(55)	(—)	(—)	(54)	(—)	(—)	(—)
	H04	0.04	0	TYP	68	76	63	—	8	82	—	—	73	—	44	21
		(1.0)			(20)	(524)	(434)	(—)	(8)	(82)	(—)	(—)	(73)	(—)	(303)	(145)
C26000	H06	0.04	0	TYP	68	86	65	—	5	88	—	—	76	—	46	—
C26130		(1.0)			(20)	(593)	(448)	(—)	(5)	(88)	(—)	(—)	(76)	(—)	(317)	(—)
	H08	0.04	0	TYP	68	94	65	—	3	91	—	—	77	—	48	23
		(1.0)			(20)	(648)	(448)	(—)	(3)	(91)	(—)	(—)	(77)	(—)	(331)	(159)
	H10	0.04	0	TYP	68	99	65	—	3	93	—	—	78	—	—	—
		(1.0)			(20)	(683)	(448)	(—)	(3)	(93)	(—)	(—)	(78)	(—)	(—)	(—)
	OS015	0.04	0	TYP	68	53	22	—	54	—	—	78	43	—	35	14
		(1.0)			(20)	(365)	(152)	(—)	(54)	(—)	(—)	(78)	(43)	(—)	(241)	(97)
	OS025	0.04	0	TYP	68	51	19	—	55	—	—	72	36	—	—	—
		(1.0)			(20)	(352)	(131)	(—)	(55)	(—)	(—)	(72)	(36)	(—)	(—)	(—)
	OS035	0.04	0	TYP	68	49	17	—	57	—	—	68	31	—	34	14
		(1.0)			(20)	(338)	(117)	(—)	(57)	(—)	(—)	(68)	(31)	(—)	(234)	(97)
	OS050	0.04	0	TYP	68	47	15	—	62	—	—	64	26	—	—	—
		(1.0)			(20)	(324)	(103)	(—)	(62)	(—)	(—)	(64)	(26)	(—)	(—)	(—)

	OS070	0.04 (1.0)	0	TYP	68 (20)	46 (317)	14 (97)	— (—)	65 (65)	— (—)	— (—)	58 (58)	15 (15)	— (—)	32 (221)	13 (90)
	OS100	0.04 (1.0)	0	TYP	68 (20)	44 (303)	11 (76)	— (—)	66 (66)	— (—)	— (—)	54 (54)	11 (11)	— (—)	— (—)	13 (90)
	M20	0.04 (1.0)	0	TYP	68 (20)	54 (372)	21 (145)	— (—)	45 (45)	— (—)	— (—)	85 (85)	49 (49)	— (—)	40 (276)	— (—)
C28000	H00	0.04 (1.0)	0	TYP	68 (20)	60 (414)	35 (241)	— (—)	30 (30)	55 (55)	— (—)	— (—)	54 (54)	— (—)	42 (290)	— (—)
	H02	0.04 (1.0)	0	TYP	68 (20)	70 (483)	50 (345)	— (—)	10 (10)	75 (75)	— (—)	— (—)	67 (67)	— (—)	44 (303)	— (—)
	M20	0.04 (1.0)	0	TYP	68 (20)	54 (372)	21 (145)	— (—)	45 (45)	— (—)	— (—)	85 (85)	49 (49)	— (—)	— (—)	— (—)
	O60	0.04 (1.0)	0	TYP	68 (20)	54 (372)	21 (145)	— (—)	45 (45)	— (—)	— (—)	80 (80)	46 (46)	— (—)	— (—)	— (—)
C50725	H02	0.01 (0.25)	0	TYP	68 (20)	74 (512)	73 (503)	— (—)	12 (12)	— (—)	— (—)	— (—)	— (—)	169 (169)	— (—)	— (—)
	H04	0.0 (0.0)	0	TYP	68 (20)	87 (596)	85 (588)	— (—)	9 (9)	— (—)	— (—)	— (—)	— (—)	193 (193)	— (—)	— (—)
	H06	0.0 (0.0)	0	TYP	68 (20)	94 (645)	92 (632)	— (—)	9 (9)	— (—)	— (—)	— (—)	— (—)	206 (206)	— (—)	— (—)
C50725	O60	0.0 (0.0)	0	TYP	68 (20)	56 (385)	38 (262)	— (—)	41 (41)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C51000	H04	0.04 (1.0)	0	TYP	68 (20)	81 (558)	75 (517)	— (—)	10 (10)	87 (87)	— (—)	— (—)	75 (75)	— (—)	— (—)	25 (172)
	H08	0.04 (1.0)	0	TYP	68 (20)	100 (689)	80 (552)	— (—)	4 (4)	95 (95)	— (—)	— (—)	79 (79)	— (—)	— (—)	22 (152)
	H10	0.04 (1.0)	0	TYP	68 (20)	107 (738)	80 (552)	— (—)	3 (3)	97 (97)	— (—)	— (—)	80 (80)	— (—)	— (—)	— (—)
	HR04	0.0 (0.0)	0	TYP	68 (20)	84 (579)	— (—)	74 (510)	14 (14)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)

Table 4 (*Continued*)

UNS Alloy	Temper	Section Size, in. (mm)	Cold Work (%)	Typ/ Min	Temp °F (°C)	Tensile Strength ksi (MPa)	Yield Strength (0.5% ext. under load) ksi (MPa)	Yield Strength (0.2% offset) ksi (MPa)	El %	Rockwell Hardness				Vickens Hard. 500 kg	Shear Strength ksi (MPa)	Fatigue Strength* ksi (MPa)
										B	C	F	30T			
C51100	H01	0.04 (1.0)	0	TYP	68 (20)	55 (379)	50 (345)	43 (296)	36 (36)	48 (48)	— (—)	— (—)	45 (45)	— (—)	— (—)	— (—)
	H04	0.04 (1.0)	0	TYP	68 (20)	80 (552)	74 (510)	77 (531)	7 (7)	86 (86)	— (—)	— (—)	74 (74)	— (—)	— (—)	— (—)
	H08	0.04 (1.0)	0	TYP	68 (20)	98 (676)	80 (552)	95 (655)	3 (3)	93 (93)	— (—)	— (—)	79 (79)	— (—)	— (—)	— (—)
	HR02	0.0 (0.0)	0	TYP	68 (20)	63 (434)	— (—)	51 (352)	26 (26)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	HR04	0.0 (0.0)	0	TYP	68 (20)	80 (552)	— (—)	73 (503)	15 (15)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	HR08	0.0 (0.0)	0	TYP	68 (20)	98 (676)	— (—)	88 (607)	8 (8)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	OS015	0.04 (1.0)	0	TYP	68 (20)	51 (352)	— (—)	23 (159)	46 (46)	— (—)	— (—)	76 (76)	— (—)	— (—)	— (—)	— (—)
	OS025	0.04 (1.0)	0	TYP	68 (20)	50 (345)	— (—)	21 (145)	46 (46)	— (—)	— (—)	75 (75)	— (—)	— (—)	— (—)	— (—)
	OS035	0.04 (1.0)	0	TYP	68 (20)	48 (331)	— (—)	19 (131)	47 (47)	— (—)	— (—)	73 (73)	— (—)	— (—)	— (—)	— (—)
	OS050	0.04 (1.0)	0	TYP	68 (20)	46 (317)	— (—)	16 (110)	48 (48)	— (—)	— (—)	70 (70)	— (—)	— (—)	— (—)	— (—)
C51900	H02	0.0 (0.0)	0	TYP	68 (20)	72 (496)	— (—)	63 (434)	25 (25)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H04	0.0050 (0.127)	0	TYP	68 (20)	88 (607)	— (—)	85 (586)	13 (13)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C51900	H08	0.0 (0.0)	0	TYP	68 (20)	107 (738)	— (—)	103 (710)	4 (4)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H10	0.0 (0.0)	0	TYP	68 (20)	111 (765)	— (—)	106 (731)	2 (2)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	HR04	0.0 (0.0)	0	TYP	68 (20)	88 (607)	— (—)	77 (531)	17 (17)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)

	HR08	0.0 (0.0)	0	TYP	68 (20)	107 (738)	— (—)	95 (655)	10 (10)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	HR10	0.0 (0.0)	0	TYP	68 (20)	111 (765)	— (—)	97 (669)	9 (9)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	OS010	0.0050 (0.127)	0	TYP	68 (20)	55 (379)	— (—)	— (—)	45 (45)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	OS015	0.0 (0.0)	0	TYP	68 (20)	56 (386)	— (—)	29 (200)	45 (45)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	OS025	0.0 (0.0)	0	TYP	68 (20)	53 (365)	— (—)	28 (193)	56 (56)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	OS035	0.0 (0.0)	0	TYP	68 (20)	52 (358)	— (—)	27 (186)	61 (61)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	OS050	0.0 (0.0)	0	TYP	68 (20)	50 (345)	— (—)	26 (179)	66 (66)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C52100	H02	0.04 (1.0)	0	TYP	68 (20)	76 (524)	55 (379)	— (—)	32 (32)	84 (84)	— (—)	— (—)	73 (73)	— (—)	— (—)	— (—)
	H04	0.04 (1.0)	0	TYP	68 (20)	93 (641)	72 (496)	— (—)	10 (10)	93 (93)	— (—)	— (—)	78 (78)	— (—)	— (—)	22 (152)
	H08	0.04 (1.0)	0	TYP	68 (20)	112 (772)	— (—)	— (—)	3 (3)	98 (98)	— (—)	— (—)	81 (81)	— (—)	— (—)	— (—)
	H10	0.04 (1.0)	0	TYP	68 (20)	120 (827)	— (—)	— (—)	2 (2)	100 (100)	— (—)	— (—)	82 (82)	— (—)	— (—)	— (—)
	HR02	0.0 (0.0)	0	TYP	68 (20)	77 (531)	— (—)	62 (427)	39 (39)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	HR04	0.0 (0.0)	0	TYP	68 (20)	93 (641)	— (—)	80 (552)	23 (23)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	HR08	0.0 (0.0)	0	TYP	68 (20)	112 (772)	— (—)	102 (703)	11 (11)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C52100	OS015	0.04 1.0	0	TYP	68 20	62 427	— —	— —	60 60	— —	— —	85 85	— —	— —	— —	— —
	OS025	0.04 (1.0)	0	TYP	68 (20)	60 (414)	24 (165)	— (—)	63 (63)	50 (50)	— (—)	82 (82)	— (—)	— (—)	— (—)	— (—)
	OS035	0.04 (1.0)	0	TYP	68 (20)	58 (400)	— (—)	— (—)	65 (65)	— (—)	— (—)	80 (80)	— (—)	— (—)	— (—)	— (—)

Table 4 (*Continued*)

UNS Alloy	Temper	Section Size, in. (mm)	Cold Work (%)	Typ/ Min	Temp °F (°C)	Tensile Strength ksi (MPa)	Yield Strength (0.5% ext. under load) ksi (MPa)	Yield Strength (0.2% offset) ksi (MPa)	El %	Rockwell Hardness				Vickens Hard. 500 kg	Shear Strength ksi (MPa)	Fatigue Strength* ksi (MPa)
										B	C	F	30T			
	OS050	0.04 (1.0)	0	TYP	68 (20)	55 (379)	— (—)	— (—)	70 (70)	— (—)	— (—)	75 (75)	— (—)	— (—)	— (—)	— (—)
C66430	H02	0.0 (0.0)	0	TYP	68 (20)	78 (538)	— (—)	70 (483)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H02	0.0 (0.0)	0	MIN	68 (20)	70 (483)	— (—)	63 (434)	15 (15)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H02	0.0 (0.0)	0	TYP	68 (20)	78 (538)	— (—)	70 (483)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H04	0.0 (0.0)	0	MIN	68 (20)	85 (586)	— (—)	79 (545)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H04	0.0 (0.0)	0	TYP	68 (20)	93 (641)	— (—)	86 (593)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H06	0.0 (0.0)	0	MIN	68 (20)	97 (669)	— (—)	90 (621)	3 (3)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H06	0.0 (0.0)	0	TYP	68 (20)	217 (1496)	— (—)	97 (669)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H08	0.0 (0.0)	0	MIN	68 (20)	105 (724)	— (—)	97 (669)	2 (2)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H08	0.0 (0.0)	0	TYP	68 (20)	— (—)	— (—)	104 (717)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H08	0.02 (0.5)	0	TYP	68 (20)	97 (669)	— (—)	90 (620)	6 (6)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H10	0.0 (0.0)	0	MIN	68 (20)	110 (758)	— (—)	101 (696)	1 (1)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H10	0.0 (0.0)	0	TYP	68 (20)	116 (800)	— (—)	108 (745)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C70260	H04	0.02 (0.5)	0	TYP	68 (20)	70 (483)	— (—)	68 (469)	4 (4)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C70260	H08	0.02 (0.5)	0	TYP	68 (20)	97 (669)	— (—)	90 (620)	6 (6)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)

C70280	TM04	0.0 (0.0)	0	MIN	68 (20)	70 (483)	— (—)	— (—)	15 (15)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TM04	0.0 (0.0)	0	TYP	68 (20)	— (—)	— (—)	65 (448)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TM06	0.0 (0.0)	0	MIN	68 (20)	80 (551)	— (—)	— (—)	10 (10)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TM06	0.0 (0.0)	0	TYP	68 (20)	— (—)	— (—)	75 (517)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TM08	0.0 (0.0)	0	MIN	68 (20)	90 (620)	— (—)	— (—)	7 (7)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TM08	0.0 (0.0)	0	TYP	68 (20)	— (—)	— (—)	90 (620)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C70290	TM04	0.0 (0.0)	0	MIN	68 (20)	80 (52)	— (—)	— (—)	15 (15)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TM04	0.0 0.0	0	TYP	68 20	— —	— —	75 517	— —	— —	— —	— —	— —	— —	— —	— —
	TM06	0.0 (0.0)	0	MIN	68 (20)	90 (620)	— (—)	— (—)	10 (10)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TM06	0.0 (0.0)	0	TYP	68 (20)	— (—)	— (—)	85 (586)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TM08	0.0 (0.0)	0	MIN	68 (20)	95 (655)	— (—)	— (—)	7 (7)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C71000	H01	0.04 (1.0)	0	TYP	68 (20)	60 (414)	48 (331)	49 (338)	20 (20)	58 (58)	— (—)	92 (92)	— (—)	— (—)	— (—)	— (—)
	H04	0.04 (1.0)	0	TYP	68 (20)	75 (517)	71 (490)	72 (496)	5 (5)	80 (80)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H08	0.04 (1.0)	0	TYP	68 (20)	82 (565)	78 (538)	79 (545)	3 (3)	84 (84)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	OS025	0.04 (1.0)	0	TYP	68 (20)	53 (365)	16 (110)	16 (110)	35 (35)	30 (30)	— (—)	75 (75)	— (—)	— (—)	— (—)	— (—)
	OS035	0.04 (1.0)	0	TYP	68 (20)	52 (359)	14 (97)	14 (97)	35 (35)	27 (27)	— (—)	73 (73)	— (—)	— (—)	— (—)	— (—)

Table 4 (*Continued*)

UNS Alloy	Temper	Section Size, in. (mm)	Cold Work (%)	Typ/ Min	Temp °F (°C)	Tensile Strength ksi (MPa)	Yield Strength (0.5% ext. under load) ksi (MPa)	Yield Strength (0.2% offset) ksi (MPa)	El %	Rockwell Hardness B	Rockwell Hardness C	Rockwell Hardness F	Rockwell Hardness 30T	Vickens Hard. 500 kg	Shear Strength ksi (MPa)	Fatigue Strength* ksi (MPa)
C71000	OS050	0.04 (1.0)	0	TYP	68 (20)	51 (352)	13 (90)	13 (90)	35 (35)	25 (25)	— (—)	72 (72)	— (—)	— (—)	— (—)	— (—)
C71500	M20	1.0 (25.4)	0	TYP	68 (20)	55 (379)	20 (138)	— (—)	45 (45)	35 (35)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C75200	H01	0.04 (1.0)	0	TYP	68 (20)	65 (448)	50 (345)	— (—)	20 (20)	73 (73)	— (—)	— (—)	65 (65)	— (—)	— (—)	— (—)
	H02	0.04 (1.0)	0	TYP	68 (20)	74 (510)	62 (427)	— (—)	8 (8)	83 (83)	— (—)	— (—)	72 (72)	— (—)	— (—)	— (—)
	H04	0.04 (1.0)	0	TYP	68 (20)	85 (586)	74 (510)	— (—)	3 (3)	87 (87)	— (—)	— (—)	75 (75)	— (—)	— (—)	— (—)
	OS015	0.04 (1.0)	0	TYP	68 (20)	60 (414)	30 (207)	— (—)	32 (32)	55 (55)	— (—)	90 (90)	— (—)	— (—)	— (—)	— (—)
	OS035	0.04 (1.0)	0	TYP	68 (20)	58 (400)	25 (172)	— (—)	40 (40)	40 (40)	— (—)	85 (85)	— (—)	— (—)	— (—)	— (—)
C76200	H06	0.0 (0.0)	0	TYP	68 (20)	108 (745)	— (—)	102 (703)	2 (2)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H08	0.0 (0.0)	0	TYP	68 (20)	115 (793)	— (—)	111 (765)	1 (1)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H10	0.0 (0.0)	0	TYP	68 (20)	119 (820)	— (—)	116 (800)	1 (1)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	OS015	0.0 (0.0)	0	TYP	68 (20)	62 (427)	— (—)	35 (241)	40 (40)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	OS035	0.0 (0.0)	0	TYP	68 (20)	57 (393)	— (—)	32 (221)	41 (41)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	OS050	0.0 (0.0)	0	TYP	68 (20)	55 (379)	— (—)	30 (207)	42 (42)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)

*Fatigue strength: 100 × 10^6 cycles unless indicated as [N] × 10^6.

others), and copper–iron–tin–zinc–magnesium alloys (C19800). In the hardened condition, the alloys exhibit yield strengths between about 50 and 70 ksi (345 and 483 MPa) and conductivities between 60 and 80% IACS.

There are a number of alloys in the conductivity range from 25 to 50% IACS, including certain beryllium coppers, brasses, tin brasses, phosphor bronzes, and copper–silicon alloys. "Red" or high-conductivity beryllium coppers such as C17410 and C17510 are at the top of this class, with yield strengths between 60 and 110 ksi (414 and 758 MPa) and conductivities in the 45–48% range.

Finally, there is a group of alloys that provides very high strength with somewhat limited electrical conductivity, typically in the range from less than 10 to nearly 25% IACS. These compositions include the high strength or "gold" beryllium coppers (C17200 and others) and copper–nickel–tin alloys such as C72600, which can be strengthened by precipitation hardening or by complex metallurgical reactions such as spinodal decomposition.

Connector alloys are an especially fertile field for copper alloy development, and new compositions offering combinations of electrical and mechanical properties tailored for specific applications are continually being brought to market. Descriptions of such alloys, as well as extensive information about the selection of other copper alloys for electrical and electronic connectors can be found at http://connector.org. Information regarding proprietary alloys offered by U.S. manufacturers is available at web sites such as:

http://www.wieland.com/
http://www.waterburyrollingmills.com/
http://www.scottbrass.com/
http://www.reverecopper.com/
http://www.ipmx.com/, http://www.outokumpu.com/
http://www.olinbrass.com/
http://www.ngkmetals.com/
http://www.themillerco.com/
http://www.husseycopper.com/
http://www.heyco-metals.com/
http://www.brushwellman.com/

6.3 Industrial Products

A number of coppers and copper alloys are produced in plate form for a variety of industrial applications. Copper plate is widely used for busbars. UNS C11000 is normally specified, but one of the OF coppers should be selected if welding is necessary.

Various brasses are also produced as plate, often for industrial and marine products where high corrosion resistance is needed. Muntz metal (C28000), admiralty (UNS C44300-C44500), and naval brasses (UNS C46200-C48600) are copper–zinc brasses modified by the addition of tin and/or nickel (for higher strength and improved corrosion resistance), as well as elements such as arsenic and antimony that inhibit the occurrence of a particular form of aqueous corrosion known as dezincification. Lead may be added to improve machinability.

Manganese and silicon bronzes are used in plate form where higher strength, high wear resistance, and better corrosion resistance than that found in brasses is needed. Aluminum bronzes comprise an interesting group of complex alloys, whose properties, depending on composition, range from moderate to very high strength. In addition to aluminum, these alloys usually contain combinations of iron, niobium (columbium), nickel, and manganese. Examples include UNS C61400 and C63200

Mechanical properties of alloys produced in rod, bar, shapes, and mechanical wire are listed in Table 5. Properties of alloys generally produced in sheet, strip, and plate forms are listed in Table 4. Numerous alloys are listed in both tables; properties may vary with product form.

7 TUBULAR PRODUCTS

Plumbing products, including water tube and fittings plus various other products broadly classified as commercial tube and fittings, constitute copper's second largest end use. In the United States, copper continues to account for approximately 80% of plumbing installations, largely based on its proven reliability. Interestingly, U.S. copper plumbing tube manufacturers now offer a 50-year warranty on the product. Mechanical property data for of alloys produced in tube form are listed in Table 6.

7.1 Water Tube

Plumbing tube manufactured in the United States is made from phosphorus deoxidized, high residual phosphorus (DHP) copper, UNS C12200. Copper plumbing tube is in fact made to very stringent compositional limits under which as many as a dozen or more trace elements are strictly controlled. The level of care exercised is quite remarkable in that roughly two thirds of domestic plumbing tube is made from scrap copper that has been recycled, remelted, and re-refined.

A group of products commonly referred to as commodity tube includes water tube, drainage tube, medical gas tube, and tube for air-conditioning and refrigeration (ACR) field service. Water tube, the most common of the plumbing tubes produced, refers to types K (heaviest), L (standard), and M (lightest) wall thickness schedules of ASTM B88, Specification for Seamless Copper Water Tube. Of identical composition as water tube are drainage tube, type DWV of B306, Specification for Copper Drainage Tube (DWV), and medical gas tube, types K and L (the same wall thickness as the corresponding water tube of the same types) of ASTM B819, Specification for Seamless Copper Tube for Medical Gas Systems, which are produced for specific applications. The thinner wall of DWV tube makes it more economical for use in low-pressure drainage systems, and the internal cleanliness of medical gas tube is required for systems conveying oxygen, nitrogen, nitrous oxide, medical compressed air, or other gases used in patient care, and even oxygen for other applications. Internally cleaned tube similar to medical gas tube is the commodity tube for use in connecting air-conditioning and refrigeration system equipment, produced to ASTM B280 as type ACR. With the exception of ACR tube that is sized by outside diameter, the actual outside diameter of these commodity tubes is $\frac{1}{8}$ in. (3.2 mm) larger than the nominal or standard size. Depending on the application, plumbing tube

Table 5 Mechanical Properties of Selected Rod, Bar and Mechanical Wire Alloys (Copper Development Association, Inc.)

UNS Alloy	Temper	Section Size in. (mm)	Cold Work %	Typ/ Min	Temp °F (°C)	Tensile Strength ksi (MPa)	Yield Strength (0.5% ext. under load) ksi (MPa)	Yield Strength (0.2% offset) ksi (MPa)	El %	Rockwell Hardness				Vick-ens Hard.	Brinell Hard.		Shear Strength ksi (MPa)	Fatigue Strength* ksi (MPa)	Izod Impact Strength ksi (MPa)
										B	C	F	30T	500 kg	500 kg	3000 kg			
C10100	H04	0.25	40	TYP	68	55	50	—	10	60	—	94	—	—	—	—	29	—	
		(6.35)			(20)	(379)	(345)	(—)	(10)	(60)	(—)	(94)	(—)	(—)	(—)	(—)	(200)	(—)	
	H04	1.0	35	TYP	68	48	44	—	16	47	—	87	—	—	—	—	27	17	
		(25.4)			(20)	(331)	(303)	(—)	(16)	(47)	(—)	(87)	(—)	(—)	(—)	(—)	(186)	(117)	
	H04	2.0	16	TYP	68	45	40	—	20	45	—	85	—	—	—	—	26	—	
		(51.0)			(20)	(310)	(276)	(—)	(20)	(45)	(—)	(85)	(—)	(—)	(—)	(—)	(179)	(—)	
	M20	1.0	—	TYP	68	32	10	—	55	—	—	40	—	—	—	—	22	—	
		(25.4)	(—)		(20)	(221)	(69)	(—)	(55)	(—)	(—)	(40)	(—)	(—)	(—)	(—)	(152)	(—)	
	OS050	1.0	—	TYP	68	32	10	—	55	—	—	40	—	—	—	—	22	—	
		(25.4)	(—)		(20)	(221)	(69)	(—)	(55)	(—)	(—)	(40)	(—)	(—)	(—)	(—)	(152)	(—)	
C11000	H04	0.25	40	TYP	68	55	50	—	10	60	—	94	—	—	—	—	29	—	
		(6.35)			(20)	(379)	(345)	(—)	(10)	(60)	(—)	(94)	(—)	(—)	(—)	(—)	(200)	(—)	
	H04	1.0	35	TYP	68	48	44	—	16	47	—	87	—	—	—	—	27	17	
		(25.4)			(20)	(331)	(303)	(—)	(16)	(47)	(—)	(87)	(—)	(—)	(—)	(—)	(186)	(117)	
	H04	2.0	16	TYP	68	45	40	—	20	45	—	85	—	—	—	—	26	—	
		(51.0)			(20)	(310)	(276)	(—)	(20)	(45)	(—)	(85)	(—)	(—)	(—)	(—)	(179)	(—)	
	M20	1.0	—	TYP	68	32	10	—	55	—	—	40	—	—	—	—	22	—	
		(25.4)	(—)		(20)	(221)	(69)	(—)	(55)	(—)	(—)	(40)	(—)	(—)	(—)	(—)	(152)	(—)	
	OS050	1.0	—	TYP	68	32	10	—	55	—	—	40	—	—	—	—	22	—	
		(25.4)	(—)		(20)	(221)	(69)	(—)	(55)	(—)	(—)	(40)	(—)	(—)	(—)	(—)	(152)	(—)	
C12200	H04	1.0	40	TYP	68	45	40		20	45		85					26		
		(25.4)			(20)	(310)	(276)	(—)	(20)								(179)		
C14500	H04	0.25	20	TYP	68	43	40	—	18	43	—	—	—	—	—	—	26	—	—
		(6.35)			(20)	(296)	(276)	(—)	(18)	(43)	(—)	(—)	(—)	(—)	(—)	(—)	(179)	(—)	(—)
	H04	0.25	45	TYP	68	53	49	—	10	54	—	—	—	—	—	—	29	—	—
		(6.35)			(20)	(365)	(338)	(—)	(10)	(54)	(—)	(—)	(—)	(—)	(—)	(—)	(200)	(—)	(—)
	H04	0.5	20	TYP	68	43	40	—	20	43	—	—	50	—	—	—	26	—	—
		(12.7)			(20)	(296)	(276)	(—)	(20)	(43)	(—)	(—)	(50)	(—)	(—)	(—)	(179)	(—)	(—)
	H04	0.5	35	TYP	68	48	44	—	15	48	—	—	—	—	—	—	27	—	—
		(12.7)			(20)	(331)	(303)	(—)	(15)	(48)	(—)	(—)	(—)	(—)	(—)	(—)	(186)	(—)	(—)

Table 5 **(*Continued*)**

UNS Alloy	Temper	Section Size in. (mm)	Cold Work %	Typ/ Min	Temp °F (°C)	Tensile Strength ksi (MPa)	Yield Strength (0.5% ext. under load) ksi (MPa)	Yield Strength (0.2% offset) ksi (MPa)	El %	Rockwell Hardness				Vick-ens Hard. 500 kg	Brinell Hard.		Shear Strength ksi (MPa)	Fatigue Strength* ksi (MPa)	Izod Impact Strength ksi (MPa)
										B	C	F	30T		500 kg	3000 kg			
	H04	0.5	6	TYP	68	38	30	—	26	36	—	—	44	—	—	—	25	—	—
		12.7			20	262	207	—	26	36	—	—	44	—	—	—	172	—	—
C14500	H04	1.0	35	TYP	68	48	44	—	20	48	—	—	—	—	—	—	27	—	
		(25.4)			(20)	(331)	(303)	(—)	(20)	(48)	(—)	(—)	(—)	(—)	(—)	(—)	(186)	(—)	
	H04	2.0	15	TYP	68	42	39	—	35	—	—	—	—	—	—	—	25	—	
			(51.0)			(20)	(290)	(269)	(—)	(35)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(172)	(—)
	OS015	0.5	0	TYP	68	33	11	—	46	—	—	43	—	—	—	—	22	—	
			(12.7)			(20)	(228)	(76)	(—)	(46)	(—)	(—)	(43)	(—)	(—)	(—)	(—)	(152)	(—)
	OS050	1.0	0	TYP	68	32	10	—	50	—	—	40	—	—	—	—	22	—	
			(25.4)			(20)	(221)	(69)	(—)	(50)	(—)	(—)	(40)	(—)	(—)	(—)	(—)	(152)	(—)
C14700	H00	0.5	6	TYP	68	38	30	—	26	36	—	—	44	—	—	—	25	—	—
		(12.7)	(0)		(20)	(262)	(207)	(—)	(26)	(36)	(—)	(—)	(44)	(—)	(—)	(—)	(172)	(—)	(—)
	H04	0.25	36	TYP	68	48	44	—	10	45	—	—	—	—	—	—	27	—	—
		(6.35)			(20)	(331)	(303)	(—)	(10)	(45)	(—)	(—)	(—)	(—)	(—)	(—)	(186)	(—)	(—)
	H04	0.375	56	TYP	68	57	55	—	8	—	—	—	—	—	—	—	29	—	—
		(9.52)			(20)	(393)	(379)	(—)	(8)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(200)	(—)	(—)
	H04	0.5	20	TYP	68	43	40	—	20	43	—	—	50	—	—	—	26	—	—
		(12.7)			(20)	(296)	(276)	(—)	(20)	(43)	(—)	(—)	(50)	(—)	(—)	(—)	(179)	(—)	(—)
	H04	0.5	35	TYP	68	—	—	—	—	48	—	—	—	—	—	—	27	—	—
		(12.7)			(20)	(—)	(—)	(—)	(—)	(48)	(—)	(—)	(—)	(—)	(—)	(—)	(186)	(—)	(—)
	H04	1.0	29	TYP	68	46	43	—	11	46	—	—	—	—	—	—	27	—	—
		(25.4)			(20)	(317)	(296)	(—)	(11)	(46)	(—)	(—)	(—)	(—)	(—)	(—)	(186)	(—)	(—)
	H04	1.625	25	TYP	68	42	38	—	20	—	—	—	—	—	—	—	26	—	—
		(41.3)			(20)	(290)	(262)	(—)	(20)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(179)	(—)	(—)
	H04	1.75	24	TYP	68	40	36	—	15	—	—	—	—	—	—	—	25	—	—
		(44.5)			(20)	(276)	(248)	(—)	(15)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(172)	(—)	(—)
	OS015	0.5	—	TYP	68	33	11	—	50	—	—	43	—	—	—	—	22	—	—
		(12.7)	(—)		(20)	(228)	(76)	(—)	(50)	(—)	(—)	(43)	(—)	(—)	(—)	(—)	(152)	(—)	(—)
	OS050	0.5	—	TYP	68	32	10	—	52	—	—	40	—	—	—	—	22	—	—
		(12.7)	(—)		(20)	(221)	(69)	(—)	(52)	(—)	(—)	(40)	(—)	(—)	(—)	(—)	(152)	(—)	(—)

C15000	TD02	1.25 (31.8)	17	TYP	68 (20)	60 (414)	58 (400)	— (—)	18 (18)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TD04	0.204 (5.18)	76	TYP	68 (20)	62 (427)	56 (386)	— (—)	8 (8)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TD04	0.375 (9.53)	44	TYP	68 (20)	68 (469)	64 (441)	— (—)	11 (11)	72 (72)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C15000	TD04	0.5 (12.7)	47	TYP	68 (20)	67 (462)	63 (434)	— (—)	15 (15)	72 (72)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TD04	0.625 (16.0)	31	TYP	68 (20)	64 (441)	62 (427)	— (—)	15 (15)	72 (72)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TD04	0.75 (19.0)	34	TYP	68 (20)	63 (434)	61 (421)	— (—)	15 (15)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TD04	0.875 (22.0)	52	TYP	68 (20)	62 (427)	60 (414)	— (—)	15 (15)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TD04	1.0 (25.4)	47	TYP	68 (20)	62 (427)	60 (414)	— (—)	15 (15)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C16200	H04	0.5 (12.7)	25	TYP	68 (20)	58 (400)	45 (310)	— (—)	12 (12)	65 (65)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H04	0.5 (12.7)	— (—)	TYP	68 (20)	73 (503)	69 (474)	70 (483)	9 (9)	73 (73)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	56 (386)	30 (207)	— (—)
	OS025	0.5 (12.7)	— (—)	TYP	68 (20)	36 (248)	12 (83)	— (—)	57 (57)	— (—)	— (—)	46 (46)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	OS050	0.5 (12.7)	— (—)	TYP	68 (20)	35 (241)	7 (48)	6 (47)	56 (56)	— (—)	— (—)	46 (46)	— (—)	— (—)	— (—)	— (—)	27 (186)	15 (100)	— (—)
C17000	TB00	0.0 (0.0)	— (—)	TYP	68 (20)	73 (500)	— (—)	20 (140)	20 (20)	65 (65)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TD04	0.375 (9.53)	— (—)	TYP	68 (20)	110 (758)	— (—)	75 (517)	8 (8)	— (—)	95 (95)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TD04	1.0 (25.4)	— (—)	TYP	68 (20)	105 (724)	— (—)	75 (517)	8 (8)	95 (95)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)

Table 5 (*Continued*)

UNS Alloy	Temper	Section Size in. (mm)	Cold Work %	Typ/ Min	Temp °F (°C)	Tensile Strength ksi (MPa)	Yield Strength (0.5% ext. under load) ksi (MPa)	Yield Strength (0.2% offset) ksi (MPa)	El %	Rockwell Hardness				Vick- ens Hard.	Brinell Hard.		Shear Strength ksi (MPa)	Fatigue Strength* ksi (MPa)	Izod Impact Strength ksi (MPa)
										B	C	F	30T	500 kg	500 kg	3000 kg			
	TD04	3.0	—	TYP	68	100	—	75	8	94	—	—	—	—	—	—	—	—	—
		(76.2)	(—)		(20)	(689)	(—)	(517)	(8)	(94)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	TF00	3.0	—	TYP	68	170	—	125	3	—	35	—	—	—	—	—	—	—	—
		(76.2)	(—)		(20)	(1172)	(—)	(862)	(3)	(—)	(35)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	TF00	0.0	—	TYP	68	170	—	125	—	—	35	—	—	—	—	—	—	—	—
		(0.0)	(—)		(20)	(1172)	(—)	(862)	(—)	(—)	(35)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	TH04	0.375	—	TYP	68	190	—	145	2	—	38	—	—	—	—	—	—	—	—
		(9.53)	(—)		(20)	(1310)	(—)	(1000)	(2)	(—)	(38)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	TH04	1.0	—	TYP	68	190	—	145	2	—	38	—	—	—	—	—	—	37	—
		(25.4)	(—)		(20)	(1310)	(—)	(1000)	(2)	(—)	(38)	(—)	(—)	(—)	(—)	(—)	(—)	(255)	(—)
C17000	TH04	3.0	—	TYP	68	182	—	135	4	—	36	—	—	—	—	—	—	—	—
		(76.2)	(—)		(20)	(1255)	(—)	(930)	(4)	(—)	(36)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
C17510	TB00	ROD	—	TYP	68	45	25	—	28	35	—	—	—	—	—	—	—	—	—
		ROD	(—)		(20)	(310)	(172)	(—)	(28)	(35)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	TD04	ROD	—	TYP	68	72	65	—	12	68	—	—	—	—	—	—	—	—	—
		ROD	(—)		(20)	(496)	(448)	(—)	(12)	(68)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	TF00	ROD	—	TYP	68	110	90	—	18	96	—	—	—	—	—	—	—	—	—
		ROD	(—)		(20)	(758)	(621)	(—)	(18)	(96)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	TH04	ROD	—	TYP	68	115	110	—	14	98	—	—	—	—	—	—	—	—	—
		ROD	(—)		(20)	(793)	(758)	(—)	(14)	(98)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	TB00	ROD	—	TYP	68	45	25	—	28	35	—	—	—	—	—	—	—	—	—
		ROD	(—)		(20)	(310)	(172)	(—)	(28)	(35)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	TD04	ROD	—	TYP	68	72	65	—	12	68	—	—	—	—	—	—	—	—	—
		ROD	(—)		(20)	(496)	(448)	(—)	(12)	(68)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	TF00	ROD	—	TYP	68	110	90	—	18	96	—	—	—	—	—	—	—	—	—
		ROD	(—)		(20)	(758)	(621)	(—)	(18)	(96)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	TH04	ROD	—	TYP	68	115	110	—	14	98	—	—	—	—	—	—	—	—	—
		ROD	(—)		(20)	(793)	(758)	(—)	(14)	(98)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
C18100	H04	0.04	99	TYP	68	70	—	—	10	—	—	—	—	—	—	—	—	—	—
		(1.0)			(20)	(483)	(—)	(—)	(10)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)

	H04	0.16 (4.06)	60	TYP	68 (20)	70 (483)	— (—)	63 (434)	6 (6)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H04	0.8 (20.3)	75	TYP	68 (20)	152 (1048)	— (—)	135 (931)	17 (17)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H04	0.8 (20.3)	90	TYP	68 (20)	158 (1089)	— (—)	141 (972)	17 (17)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H04	9.16 (233.0)	60	TYP	68 (20)	75 (517)	— (—)	68 (469)	11 (11)	80 (80)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C18200	TB00	0.5 (12.7)	0	TYP	68 (20)	45 (310)	14 (97)	— (—)	40 (40)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TD00	0.156 (3.96)	91	TYP	68 (20)	86 (593)	77 (531)	— (—)	14 (14)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TD00	0.5 (12.7)	60	TYP	68 (20)	57 (393)	56 (386)	— (—)	11 (11)	65 (65)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C18200	TB00	0.5 (12.7)	0	TYP	68 (20)	45 (310)	14 (97)	— (—)	40 (40)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TD00	0.156 (3.96)	91	TYP	68 (20)	86 (593)	77 (531)	— (—)	14 (14)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TD00	0.5 (12.7)	60	TYP	68 (20)	57 (393)	56 (386)	— (—)	11 (11)	65 (65)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TB00	0.5 (12.7)	0	TYP	68 (20)	45 (310)	14 (97)	— (—)	40 (40)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TD00	0.156 (3.96)	91	TYP	68 (20)	86 (593)	77 (531)	— (—)	14 (14)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TD00	0.5 (12.7)	60	TYP	68 (20)	57 (393)	56 (386)	— (—)	11 (11)	65 (65)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TB00	0.5 (12.7)	0	TYP	68 (20)	45 (310)	14 (97)	— (—)	40 (40)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C18400	TB00	0.5 (12.7)	0	TYP	68 (20)	45 (310)	14 (97)	— (—)	40 (40)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)

Table 5 (*Continued*)

UNS Alloy	Temper	Section Size in. (mm)	Cold Work %	Typ/ Min	Temp °F (°C)	Tensile Strength ksi (MPa)	Yield Strength (0.5% ext. under load) ksi (MPa)	Yield Strength (0.2% offset) ksi (MPa)	El %	Rockwell Hardness				Vick-ens Hard. 500 kg	Brinell Hard.		Shear Strength ksi (MPa)	Fatigue Strength* ksi (MPa)	Izod Impact Strength ksi (MPa)
										B	C	F	30T		500 kg	3000 kg			
	TD00	0.156 (3.96)	91	TYP	68 (20)	86 (593)	77 (531)	— (—)	14 (14)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TD00	0.5 (12.7)	60	TYP	68 (20)	57 (393)	56 (386)	— (—)	11 (11)	65 (65)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TF00	0.5 (12.7)	0	TYP	68 (20)	70 (483)	55 (379)	— (—)	21 (21)	70 (70)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TF00	1.0 (25.4)	0	TYP	68 (20)	72 (496)	65 (448)	— (—)	18 (18)	80 (80)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TF00	2.0 (51.0)	0	TYP	68 (20)	70 (483)	65 (448)	— (—)	18 (18)	75 (75)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TF00	3.0 76.0	0	TYP	68 20	65 448	55 379	— —	18 18	70 70	— —	— —	— —	— —	— —	— —	— —	— —	— —
	TF00	4.0 (102.0)	0	TYP	68 (20)	55 (379)	43 (296)	— (—)	25 (25)	68 (68)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TH01	0.156 (3.96)	90	TYP	68 (20)	74 (510)	73 (503)	— (—)	5 (5)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TH01	0.5 (12.7)	50	TYP	68 (20)	77 (531)	67 (462)	— (—)	19 (19)	83 (83)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C19100	HR01	0.125 (3.18)	75	TYP	68 (20)	104 (717)	77 (531)	92 (634)	6 (6)	95 (95)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	56 (386)	— (—)	— (—)
	HR01	0.25 (6.35)	35	TYP	68 (20)	84 (579)	73 (503)	77 (531)	10 (10)	85 (85)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	44 (303)	33 (228)	— (—)
	HR01	0.375 (9.53)	—	TYP	68 (20)	94 (648)	75 (517)	86 (593)	10 (10)	90 (90)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	48 (331)	— (—)	— (—)
	HR01	0.5 (12.7)	— (—)	TYP	68 (20)	80 (552)	70 (483)	73 (503)	15 (15)	85 (85)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	43 (296)	— (—)	— (—)
	HR01	1.0 (25.4)	— (—)	TYP	68 (20)	78 (538)	68 (469)	70 (483)	27 (27)	84 (84)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	41 (283)	33 (228)	— (—)
C23000	H00	0.08 (2.0)	— (—)	TYP	68 (20)	50 (345)	— (—)	— (—)	25 (25)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	35 (241)	20 (138)	— (—)

	H01	0.08	—	TYP	68	59	—	—	11	—	—	—	—	—	—	—	38	—	—
		(2.0)	(—)		(20)	(407)	(—)	(—)	(11)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(262)	(—)	(—)
	H02	0.08	—	TYP	68	72	—	—	8	—	—	—	—	—	—	—	43	—	—
		(2.0)	(—)		(20)	(496)	(—)	(—)	(8)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(296)	(—)	(—)
	H04	0.08	—	TYP	68	88	—	—	6	—	—	—	—	—	—	—	48	—	—
		(2.0)	(—)		(20)	(607)	(—)	(—)	(6)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(331)	(—)	(—)
	H08	0.08	—	TYP	68	105	—	—	—	—	—	—	—	—	—	—	54	—	—
		(2.0)	(—)		(20)	(724)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(372)	(—)	(—)
	OS015	0.08	—	TYP	68	45	—	—	—	—	—	—	—	—	—	—	33	—	—
		2.0	—		20	310	—	—	—	—	—	—	—	—	—	—	228	—	—
	OS025	0.08	—	TYP	68	43	—	—	—	—	—	—	—	—	—	—	32	—	—
		(2.0)	(—)		(20)	(296)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(221)	(—)	(—)
	OS035	0.08	—	TYP	68	41	—	—	48	—	—	—	—	—	—	—	31	—	—
		(2.0)			(20)	(283)	(—)	(—)	(48)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(214)	(—)	(—)
C26000	H00	1.0	6	TYP	68	55	40	—	48	60	—	—	—	—	—	—	36	—	—
		(25.4)			(20)	(379)	(276)	(—)	(48)	(60)	(—)	(—)	(—)	(—)	(—)	(—)	(248)	(—)	(—)
	H04	1.0	20	TYP	68	70	52	—	30	80	—	—	—	—	—	—	42	22	—
		(25.4)			(20)	(483)	(359)	(—)	(30)	(80)	(—)	(—)	(—)	(—)	(—)	(—)	(290)	(152)	(—)
	OS050	1.0	0	TYP	68	48	16	—	65	—	—	65	—	—	—	—	34	—	—
		(25.4)			(20)	(331)	(110)	(—)	(65)	(—)	(—)	(65)	(—)	(—)	(—)	(—)	(234)	(—)	(—)
	H00	1.0	6	TYP	68	55	40	—	48	60	—	—	—	—	—	—	36	—	—
		(25.4)			(20)	(379)	(276)	(—)	(48)	(60)	(—)	(—)	(—)	(—)	(—)	(—)	(248)	(—)	(—)
C26000	H04	1.0	20	TYP	68	70	52	—	30	80	—	—	—	—	—	—	42	22	—
		(25.4)			(20)	(483)	(359)	(—)	(30)	(80)	(—)	(—)	(—)	(—)	(—)	(—)	(290)	(152)	(—)
	OS050	1.0	—	TYP	68	48	16	—	65	—	—	65	—	—	—	—	34	—	—
		(25.4)			(20)	(331)	(110)	(—)	(65)	(—)	(—)	(65)	(—)	(—)	(—)	(—)	(234)	(—)	(—)
	H00	0.08	0	TYP	68	58	—	—	35	—	—	—	—	—	—	—	38	—	—
		(2.0)			(20)	(400)	(—)	(—)	(35)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(262)	(—)	(—)
	H01	0.08	—	TYP	68	70	—	—	20	—	—	—	—	—	—	—	—	—	—
		(2.0)			(20)	(483)	(—)	(—)	(20)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)

Table 5 (*Continued*)

UNS Alloy	Temper	Section Size in. (mm)	Cold Work %	Typ/ Min	Temp °F (°C)	Tensile Strength ksi (MPa)	Yield Strength (0.5% ext. under load) ksi (MPa)	Yield Strength (0.2% offset) ksi (MPa)	El %	Rockwell Hardness				Vick-ens Hard.	Brinell Hard.		Shear Strength ksi (MPa)	Fatigue Strength* ksi (MPa)	Izod Impact Strength ksi (MPa)
										B	C	F	30T	500 kg	500 kg	3000 kg			
	H06	0.08 (2.0)	—	TYP	68 (20)	124 (855)	— (—)	— (—)	4 (4)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H08	0.08 (2.0)	—	TYP	68 (20)	130 (896)	— (—)	— (—)	3 (3)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	60 (414)	22 (152)	— (—)
	OS015	0.08 (2.0)	—	TYP	68 (20)	54 (372)	— (—)	— (—)	56 (56)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	OS025	0.08 (2.0)	—	TYP	68 (20)	52 (359)	— (—)	— (—)	58 (58)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	OS035	0.08 (2.0)	—	TYP	68 (20)	50 (345)	— (—)	— (—)	60 (60)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	34 (234)	— (—)	— (—)
	OS050	0.08 (2.0)	—	TYP	68 (20)	48 (331)	— (—)	— (—)	64 (64)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C27000	H04	1.0 (25.4)	6	TYP	68 (20)	55 (379)	40 (276)	— (—)	48 (48)	55 (55)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	36 (248)	— (—)	— (—)
	OS050	1.0 (25.4)	0	TYP	68 (20)	48 (331)	16 (110)	— (—)	65 (65)	— (—)	— (—)	65 (65)	— (—)	— (—)	— (—)	— (—)	34 (234)	— (—)	— (—)
C27000	H00	0.08 (2.0)	—	TYP	68 (20)	58 (400)	— (—)	— (—)	35 (35)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	38 (262)	— (—)	— (—)
	H01	0.08 (2.0)	—	TYP	68 (20)	70 (483)	— (—)	— (—)	20 (20)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	42 (290)	22 (152)	— (—)
	H02	0.08 (2.0)	—	TYP	68 (20)	88 (607)	— (—)	— (—)	15 (15)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H04	0.08 (2.0)	—	TYP	68 (20)	110 (758)	— (—)	— (—)	8 (8)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	55 (379)	— (—)	— (—)
	H06	0.08 (2.0)	—	TYP	68 (20)	120 (827)	— (—)	— (—)	4 (4)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	60 (414)	— (—)	— (—)
C27000	H08	0.08 (2.0)	—	TYP	68 (20)	128 (883)	— (—)	— (—)	3 (3)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	60 (414)	— (—)	— (—)
	OS035	0.08 (2.0)	—	TYP	68 (20)	50 (345)	— (—)	— (—)	60 (60)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	34 (234)	— (—)	— (—)

C28000	H01	1.0	—	TYP	68	72	50	—	25	78	—	—	—	—	—	—	45	—	—
		(25.4)			(20)	(496)	(345)	(—)	(25)	(78)	(—)	(—)	(—)	(—)	(—)	(—)	(310)	(—)	(—)
	M30	1.0	—	TYP	68	52	20	—	52	—	—	78	—	—	—	—	39	—	—
		(25.4)			(20)	(359)	(138)	(—)	(52)	(—)	(—)	(78)	(—)	(—)	(—)	(—)	(269)	(—)	(—)
	O60	1.0	—	TYP	68	54	21	—	50	—	—	80	—	—	—	—	40	—	—
		(25.4)			(20)	(372)	(145)	(—)	(50)	(—)	(—)	(80)	(—)	(—)	(—)	(—)	(276)	(—)	(—)
C34500	H01	1.0	—	MAX	68	65	—	—	—	—	—	—	—	—	—	—	—	—	—
		(12.2)			(20)	(448)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	H01	1.0	—	MIN	68	50	25	—	10	—	—	—	—	—	—	—	—	—	—
		(12.2)			(20)	(345)	(172)	(—)	(10)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	H01	1.0	—	MAX	68	124	—	—	—	145	—	—	—	—	—	—	—	—	—
		(25.4)			(20)	(854)	(—)	(—)	(—)	(145)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	H01	1.0	—	MIN	68	92	35	—	35	95	—	—	—	—	—	—	—	—	—
		(25.4)			(20)	(635)	(241)	(—)	(35)	(95)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	H02	1.0	—	MAX	68	80	—	—	—	—	—	—	—	—	—	—	—	—	—
		(12.2)			(20)	(552)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	H02	1.0	—	MIN	68	57	25	—	7	—	—	—	—	—	—	—	—	—	—
		(12.2)			(20)	(393)	(172)	(—)	(7)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	H02	1.0	—	MAX	68	132	—	—	—	155	—	—	—	—	—	—	—	—	—
		(25.4)			(20)	(910)	(—)	(—)	(—)	(155)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	H02	1.0	—	MIN	68	105	45	—	25	115	—	—	—	—	—	—	—	—	—
		(25.4)			(20)	(724)	(310)	(—)	(25)	(115)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	O60	0.5	—	MIN	68	46	16	—	20	—	—	—	—	—	—	—	—	—	—
		(12.2)			(20)	(317)	(110)	(—)	(20)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	O60	1.0	—	MAX	68	—	—	—	—	90	—	—	—	—	—	—	—	—	—
		(25.4)			(20)	(—)	(—)	(—)	(—)	(90)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	O60	1.0	—	MIN	68	84	30	—	55	—	—	—	—	—	—	—	—	—	—
		(25.4)			(20)	(579)	(206)	(—)	(55)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
C35300	H04	1.0	20	TYP	68	58	45	—	25	75	—	—	—	—	—	—	—	—	—
		(25.4)			(20)	(400)	(310)	(—)	(25)	(75)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)

Table 5 (*Continued*)

UNS Alloy	Temper	Section Size in. (mm)	Cold Work %	Typ/ Min	Temp °F (°C)	Tensile Strength ksi (MPa)	Yield Strength (0.5% ext. under load) ksi (MPa)	Yield Strength (0.2% offset) ksi (MPa)	El %	Rockwell Hardness				Vick-ens Hard. 500 kg	Brinell Hard.		Shear Strength ksi (MPa)	Fatigue Strength* ksi (MPa)	Izod Impact Strength ksi (MPa)
										B	C	F	30T		500 kg	3000 kg			
C36000 (BAR)	H02	0.5 (12.7)	—	MIN	68 (20)	50 (345)	25 (170)	— (—)	10 (10)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H02	2.0 (50.8)	—	MIN	68 (20)	85 (585)	32 (220)	— (—)	35 (35)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H20	2.01 (51.1)	—	MIN	68 (20)	40 (275)	15 (105)	— (—)	20 (20)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	O60	1.0 (25.4)	—	MIN	68 (20)	44 (305)	12 (125)	— (—)	20 (20)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C36000 (ROD)	H02	0.5 (51.0)	—	TYP	68 (20)	57 (395)	25 (170)	— (—)	7 (7)	75 (75)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	32 (221)	20 (138)	— (—)
	H02	1.0 (25.4)	—	MIN	68 (20)	55 (380)	44 (303)	— (—)	25 (25)	75 (75)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	34 (234)	— (—)	— (—)
	H02	2.0 (50.8)	—	MIN	68 (20)	50 (345)	20 (140)	— (—)	15 (15)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H02	4.0 (101.6)	—	MIN	68 (20)	45 (310)	15 (105)	— (—)	20 (20)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H04	0.1875 (6.35)	—	MIN	68 (20)	80 (550)	45 (310)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H04	0.1875 (6.35)	—	TYP	68 (20)	— (—)	— (—)	— (—)	— (—)	80 (80)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	38 (262)	— (—)	— (—)
	H04	0.5 (12.7)	—	MIN	68 (20)	70 (480)	35 (240)	— (—)	4 (25)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H04	0.5 (12.7)	—	TYP	68 (20)	— (—)	— (—)	— (—)	— (—)	78 (78)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	34 (234)	— (—)	— (—)
	H04	0.75 (19.1)	—	MIN	68 (20)	65 (450)	30 (205)	— (—)	6 (6)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	O60	1.0 (25.4)	—	MIN	68 (20)	48 (330)	20 (124)	— (—)	53 (53)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	O60	1.0 (25.4)	—	TYP	68 (20)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	68 (68)	— (—)	— (—)	— (—)	— (—)	30 (207)	— (—)	— (—)

	O60	2.0 (50.8)	—	MIN	68 (20)	44 (305)	18 (18)	— (—)	20 (20)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C36000 (SHAPES)	H04	0.5 (12.7)	11	TYP	68 (20)	56 (386)	45 (310)	— (—)	20 (20)	62 (62)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	33 (228)	— (—)	0.0 (0.0)
C36000 (SHAPES)	M30	0.5 (12.7)	—	TYP	68 (20)	49 (338)	18 (124)	— (—)	50 (50)	— (—)	— (—)	68 (68)	— (—)	— (—)	— (—)	— (—)	30 (207)	— (—)	— (—)
C37700 (ROD)	M30	1.0 (25.4)	—	TYP	68 (20)	52 (359)	20 (138)	— (—)	45 (45)	— (—)	— (—)	78 (78)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C37700 (SHAPES)	M30	1.0 (25.4)	—	TYP	68 (20)	52 (359)	20 (138)	— (—)	45 (45)	— (—)	— (—)	78 (78)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C46400	H01	0.25 (6.35)	10	TYP	68 (20)	70 (483)	48 (331)	— (—)	25 (25)	80 (80)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	43 (296)	— (—)	— (—)
	H01	1.0 (25.4)	8	TYP	68 (20)	69 (476)	46 (317)	— (—)	27 (27)	78 (78)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	43 (296)	— (—)	— (—)
	H01	2.0 (51.0)	8	TYP	68 (20)	67 (462)	40 (276)	— (—)	35 (35)	75 (75)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	43 (296)	— (—)	— (—)
	H02	0.25 (6.35)	20	TYP	68 (20)	80 (552)	57 (393)	— (—)	20 (20)	85 (85)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	45 (310)	— (—)	— (—)
	H02	1.0 (25.4)	20	TYP	68 (20)	75 (517)	53 (365)	— (—)	20 (20)	82 (82)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	44 (303)	— (—)	— (—)
	O50	0.25 (6.35)	0	TYP	68 (20)	63 (434)	30 (207)	— (—)	40 (40)	60 (60)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	42 (290)	— (—)	— (—)
	O50	1.0 (25.4)	0	TYP	68 (20)	63 (434)	30 (207)	— (—)	40 (40)	60 (60)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	42 (290)	— (—)	— (—)
	O50	2.0 (51.0)	0	TYP	68 (20)	62 (427)	28 (193)	— (—)	43 (43)	60 (60)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	42 (290)	— (—)	— (—)
	O60	0.25 (6.35)	0	TYP	68 (20)	58 (400)	27 (186)	— (—)	45 (45)	56 (56)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	40 (276)	— (—)	— (—)
	O60	1.0 (25.4)	0	TYP	68 (20)	57 (393)	25 (172)	— (—)	47 (47)	55 (55)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	40 (276)	— (—)	— (—)

Table 5 (*Continued*)

UNS Alloy	Temper	Section Size in. (mm)	Cold Work %	Typ/ Min	Temp °F (°C)	Tensile Strength ksi (MPa)	Yield Strength (0.5% ext. under load) ksi (MPa)	Yield Strength (0.2% offset) ksi (MPa)	El %	Rockwell Hardness				Vick-ens Hard.	Brinell Hard.		Shear Strength ksi (MPa)	Fatigue Strength* ksi (MPa)	Izod Impact Strength ksi (MPa)
										B	C	F	30T	500 kg	500 kg	3000 kg			
	O60	2.0	0	TYP	68	56	25	—	47	55	—	—	—	—	—	—	40	—	—
		51.0			20	386	172	—	47	55	—	—	—	—	—	—	276	—	—
C48200	H01	1.5	4	TYP	68	66	40	—	32	75	—	—	—	—	—	—	40	—	—
(BAR)		(38.0)			(20)	(455)	(276)	(—)	(32)	(75)	(—)	(—)	(—)	(—)	(—)	(—)	(276)	(—)	(—)
	M30	0.375	0	TYP	68	63	33	—	34	60	—	—	—	—	—	—	39	—	—
		(9.525)			(20)	(434)	(228)	(—)	(34)	(60)	(—)	(—)	(—)	(—)	(—)	(—)	(269)	(—)	(—)
C48200	H01	1.0	8	TYP	68	69	46	—	20	78	—	—	—	—	—	—	40	—	—
		(25.4)			(20)	(476)	(317)	(—)	(20)	(78)	(—)	(—)	(—)	(—)	(—)	(—)	(276)	(—)	(—)
C48200	H01	2.0	8	TYP	68	67	40	—	30	75	—	—	—	—	—	—	40	—	—
		(51.0)			(20)	(462)	(276)	(—)	(30)	(75)	(—)	(—)	(—)	(—)	(—)	(—)	(276)	(—)	(—)
	H02	1.0	20	TYP	68	75	53	—	15	82	—	—	—	—	—	—	41	—	—
		(25.4)			(20)	(517)	(365)	(—)	(15)	(82)	(—)	(—)	(—)	(—)	(—)	(—)	(283)	(—)	(—)
	H02	2.0	15	TYP	68	70	52	—	17	78	—	—	—	—	—	—	41	—	—
		(51.0)			(20)	(483)	(359)	(—)	(17)	(78)	(—)	(—)	(—)	(—)	(—)	(—)	(283)	(—)	(—)
	H04	3.0	4	TYP	68	63	33	—	43	78	—	—	—	—	—	—	40	—	—
		76.0			20	434	228	—	43	78	—	—	—	—	—	—	276	—	—
	O50	1.0	0	TYP	68	63	30	—	35	60	—	—	—	—	—	—	39	—	—
		(25.4)			(20)	(434)	(207)	(—)	(35)	(60)	(—)	(—)	(—)	(—)	(—)	(—)	(269)	(—)	(—)
	O50	2.0	0	TYP	68	62	28	—	37	60	—	—	—	—	—	—	39	—	—
		(51.0)			(20)	(427)	(193)	(—)	(37)	(60)	(—)	(—)	(—)	(—)	(—)	(—)	(269)	(—)	(—)
	O60	1.0	0	TYP	68	57	25	—	40	55	—	—	—	—	—	—	38	—	—
		(25.4)			(20)	(393)	(172)	(—)	(40)	(55)	(—)	(—)	(—)	(—)	(—)	(—)	(262)	(—)	(—)
	O60	2.0	0	TYP	68	56	25	—	40	55	—	—	—	—	—	—	38	—	—
		(51.0)			(20)	(386)	(172)	(—)	(40)	(55)	(—)	(—)	(—)	(—)	(—)	(—)	(262)	(—)	(—)
	H01	2.0	8	TYP	68	67	40	—	30	75	—	—	—	—	—	—	40	—	—
		(51.0)			(20)	(462)	(276)	(—)	(30)	(75)	(—)	(—)	(—)	(—)	(—)	(—)	(276)	(—)	(—)
C51000	H04	0.5	20	TYP	68	75	65	—	25	80	—	—	—	—	—	—	—	—	—
		(12.7)			(20)	(517)	(448)	(—)	(25)	(80)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	H04	1.0	20	TYP	68	70	58	—	25	78	—	—	—	—	—	—	—	—	—
		(25.4)			(20)	(483)	(400)	(—)	(25)	(78)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)

	H01	0.08 (2.0)	—	TYP	68 (20)	68 (469)	60 (414)	— (—)	24 (24)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H02	0.08 (2.0)	—	TYP	68 (20)	85 (586)	80 (552)	— (—)	8 (8)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H04	0.08 (2.0)	—	TYP	68 (20)	110 (758)	— (—)	— (—)	3 (3)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	27 (186)	— (—)
	OS035	0.08 (2.0)	0	TYP	68 (20)	50 (345)	20 (138)	— (—)	58 (58)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	TD04	0.08 (2.0)	75	TYP	68 (20)	130 (896)	— (—)	— (—)	3 (3)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	30 (207)	— (—)
	TD04	0.08 (2.0)	84	TYP	68 (20)	140 (965)	— (—)	— (—)	2 (2)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C52100 (ROD)	H04	0.5 (12.7)	20	TYP	68 (20)	80 (552)	65 (448)	— (—)	33 (33)	85 (85)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C52100	H01	0.08 (2.0)	—	TYP	68 (20)	81 (558)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H02	0.08 (2.0)	—	TYP	68 (20)	105 (724)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H04	0.08 (2.0)	—	TYP	68 (20)	130 (896)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H06	0.08 (2.0)	—	TYP	68 (20)	140 (965)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	OS035	0.08 (2.0)	0	TYP	68 (20)	60 (414)	24 (165)	— (—)	65 (65)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C61300	H04	0.5 (12.7)	25	TYP	68 (20)	85 (586)	58 (400)	— (—)	35 (35)	91 (91)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	48 (331)	— (—)	— (—)
	H04	1.0 (25.4)	25	TYP	68 (20)	82 (565)	55 (379)	— (—)	35 (35)	90 (90)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	45 (310)	— (—)	— (—)
	H04	2.0 (51.0)	25	TYP	68 (20)	80 (552)	48 (331)	— (—)	35 (35)	88 (88)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	40 (276)	— (—)	— (—)

Table 5 (*Continued*)

UNS Alloy	Temper	Section Size in. (mm)	Cold Work %	Typ/ Min	Temp °F (°C)	Tensile Strength ksi (MPa)	Yield Strength (0.5% ext. under load) ksi (MPa)	Yield Strength (0.2% offset) ksi (MPa)	El %	Rockwell Hardness				Vick-ens Hard. 500 kg	Brinell Hard.		Shear Strength ksi (MPa)	Fatigue Strength* ksi (MPa)	Izod Impact Strength ksi (MPa)
										B	C	F	30T		500 kg	3000 kg			
	H04	0.5 (12.7)	25	TYP	68 (20)	85 (586)	58 (400)	— (—)	35 (35)	91 (91)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	48 (331)	— (—)	— (—)
C61800	H04	1.0 (25.4)	15	TYP	68 (20)	85 (586)	43 (293)	— (—)	23 (23)	89 (89)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	47 (324)	82 (565)	— (—)
	H04	2.0 (51.0)	15	TYP	68 (20)	83 (569)	39 (269)	— (—)	25 (25)	88 (88)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	45 (310)	— (—)	— (—)
	H04	3.0 (76.0)	15	TYP	68 (20)	80 (552)	39 (269)	— (—)	28 (28)	88 (88)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	43 (296)	— (—)	— (—)
C63000 (BAR)	H04	1.0 (25.4)	10	TYP	68 (20)	110 (758)	62 (427)	— (—)	15 (15)	97 (97)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H04	2.0 (51.0)	10	TYP	68 (20)	100 (689)	60 (414)	— (—)	15 (15)	96 (96)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	M30	3.0 (76.0)	0	TYP	68 (20)	90 (621)	50 (345)	— (—)	15 (15)	96 (96)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C63000	H04	1.0 (25.4)	10	TYP	68 (20)	118 (814)	75 (517)	— (—)	15 (15)	98 (98)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	70 (483)	112 (772)	— (—)
C63000	H04	2.0 (51.0)	10	TYP	68 (20)	115 (793)	65 (448)	— (—)	18 (18)	96 (96)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	69 (476)	— (—)	— (—)
	H04	3.0 (76.0)	10	TYP	68 (20)	112 (772)	62 (427)	— (—)	20 (20)	96 (96)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	65 (448)	— (—)	— (—)
	M30	4.0 (102.0)	0	TYP	68 (20)	100 (689)	60 (414)	— (—)	15 (15)	96 (96)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	62 (427)	36 (248)	— (—)
C65500	H02	1.0 (25.4)	20	TYP	68 (20)	78 (538)	45 (310)	— (—)	35 (35)	85 (85)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	52 (359)	— (—)	— (—)
	H04	1.0 (25.4)	36	TYP	68 (20)	92 (634)	55 (379)	— (—)	22 (22)	90 (90)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	58 (400)	— (—)	— (—)
	H06	1.0 (25.4)	50	TYP	68 (20)	108 (745)	60 (414)	— (—)	13 (13)	95 (95)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	62 (427)	— (—)	— (—)
	OS050	1.0 (25.4)	0	TYP	68 (20)	58 (400)	22 (152)	— (—)	60 (60)	60 (60)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	43 (296)	— (—)	— (—)

	H00	0.08 (2.0)	0	TYP	68 (20)	70 (483)	40 (276)	— (—)	35 (35)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	48 (331)	— (—)	— (—)
	H01	0.08 (2.0)	0	TYP	68 (20)	80 (552)	48 (331)	— (—)	20 (20)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	52 (359)	— (—)	— (—)
	H02	0.08 (2.0)	0	TYP	68 (20)	98 (676)	57 (393)	— (—)	8 (8)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	58 (400)	— (—)	— (—)
	H04	0.08 (2.0)	0	TYP	68 (20)	125 (862)	65 (448)	— (—)	5 (5)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	65 (448)	29 (200)	— (—)
	H08	0.08 (2.0)	0	TYP	68 (20)	145 (1000)	70 (483)	— (—)	3 (3)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	70 (483)	30 (207)	— (—)
	OS035	0.08 (2.0)	0	TYP	68 (20)	60 (414)	25 (172)	— (—)	60 (60)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	43 (296)	— (—)	— (—)
C67000	H02	0.08 (2.0)	0	TYP	68 (20)	90 (621)	— (—)	— (—)	5 (5)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C67500	H01	1.0 (25.4)	10	TYP	68 (20)	77 (531)	45 (310)	— (—)	23 (23)	83 (83)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	47 (324)	— (—)	— (—)
	H01	2.0 (51.0)	10	TYP	68 (20)	72 (496)	42 (290)	— (—)	27 (27)	77 (77)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	44 (303)	— (—)	— (—)
	H02	1.0 (25.4)	20	TYP	68 (20)	84 (579)	60 (414)	— (—)	19 (19)	90 (90)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	48 (331)	— (—)	— (—)
C67500	O60	1.0 (25.4)	0	TYP	68 (20)	65 (448)	30 (207)	— (—)	33 (33)	65 (65)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	42 (290)	— (—)	— (—)
C71500	H04	1.0 (25.4)	20	TYP	68 (20)	75 (517)	70 (483)	— (—)	15 (15)	80 (80)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C72900	TF00	0.03 (0.8)	0	TYP	68 (20)	160 (1103)	— (—)	— (—)	4 (4)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C74500	H04	0.08 (2.0)	10	TYP	68 (20)	65 (448)	— (—)	— (—)	25 (25)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H04	0.08 (2.0)	20	TYP	68 (20)	72 (496)	— (—)	— (—)	10 (10)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H04	0.08 (2.0)	37	TYP	68 (20)	85 (586)	— (—)	— (—)	7 (7)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H04	0.08 (2.0)	60	TYP	68 (20)	105 (724)	— (—)	— (—)	5 (5)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
	H04	0.08 (2.0)	75	TYP	68 (20)	120 (827)	— (—)	— (—)	3 (3)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)

Table 5 (*Continued*)

UNS Alloy	Temper	Section Size in. (mm)	Cold Work %	Typ/Min	Temp °F (°C)	Tensile Strength ksi (MPa)	Yield Strength (0.5% ext. under load) ksi (MPa)	Yield Strength (0.2% offset) ksi (MPa)	El %	Rockwell Hardness				Vickens Hard. 500 kg	Brinell Hard.		Shear Strength ksi (MPa)	Fatigue Strength* ksi (MPa)	Izod Impact Strength ksi (MPa)
										B	C	F	30T		500 kg	3000 kg			
	H04	0.08	84	TYP	68	130	—	—	1	—	—	—	—	—	—	—	—	—	—
		(2.0)			(20)	(896)	(—)	(—)	(1)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	OS015	0.08	0	TYP	68	63	—	—	35	—	—	—	—	—	—	—	—	—	—
		(2.0)			(20)	(434)	(—)	(—)	(35)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	OS025	0.08	0	TYP	68	58	—	—	40	—	—	—	—	—	—	—	—	—	—
		(2.0)			(20)	(400)	(—)	(—)	(40)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	OS035	0.08	0	TYP	68	56	—	—	45	—	—	—	—	—	—	—	—	—	—
		(2.0)			(20)	(386)	(—)	(—)	(45)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	OS050	0.08	0	TYP	68	52	—	—	48	—	—	—	—	—	—	—	—	—	—
		(2.0)			(20)	(359)	(—)	(—)	(48)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	OS070	0.08	0	TYP	68	50	—	—	50	—	—	—	—	—	—	—	—	—	—
		(2.0)			(20)	(345)	(—)	(—)	(50)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
C75200	H04	0.5	20	TYP	68	70	60	—	20	78	—	—	—	—	—	—	—	—	—
		(12.7)			(20)	(483)	(414)	(—)	(20)	(78)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	OS035	0.5	0	TYP	68	56	25	—	42	—	—	—	—	—	—	—	—	—	—
		12.7			(20)	(386)	(172)	(—)	(42)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
		0.08	0	TYP	68	73	65	—	16	—	—	—	—	—	—	—	—	—	—
		(2.0)			(20)	(503)	(448)	(—)	(16)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
C75200	H02	0.08	—	TYP	68	86	80	—	7	—	—	—	—	—	—	—	—	—	—
		(2.0)			(20)	(593)	(552)	(—)	(7)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	H04	0.08	—	TYP	68	103	90	—	3	—	—	—	—	—	—	—	—	—	—
		(2.0)			(20)	(710)	(621)	(—)	(3)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	OS015	0.08	0	TYP	68	60	30	—	35	—	—	—	—	—	—	—	—	—	—
		(2.0)			(20)	(414)	(207)	(—)	(35)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)	(—)
	OS035	0.08	0	TYP	68	58	25	—	45	—	—	—	—	—	—	—	—	—	—
		2.0			20	400	172	—	45	—	—	—	—	—	—	—	—	—	—

*Fatigue strength: 100×10^6 cycles unless indicated as [N] $\times 10^6$.

Table 6 Mechanical Properties of Selected Tube Alloys (Copper Development Association, Inc.)

UNS Alloy	Temper	Section Size in. (mm)	Cold Work %	Typ/ Min	Temp °F (°C)	Tensile Strength ksi (MPa)	Yield Strength (0.5% ext. under load) ksi (MPa)	El %	Rockwell Hardness				Shear Strength ksi (MPa)
									B	C	F	30T	
C10100	H55	0.065	15	TYP	68	40	32	25	35	—	77	45	26
C11000		(1.65)			(20)	(276)	(221)	(25)	(35)	(—)	(77)	(45)	(179)
	H80	0.065	40	TYP	68	55	50	8	60	—	95	63	29
		(1.65)			(20)	(379)	(345)	(8)	(60)	(—)	(95)	(63)	(200)
	OS025	0.065	0	TYP	68	34	11	45	—	—	45	—	23
		(1.65)			(20)	(234)	(76)	(45)	(—)	(—)	(45)	(—)	(159)
	OS050	0.065	0	TYP	68	32	10	45	—	—	40	—	22
		(1.65)			(20)	(221)	(69)	(45)	(—)	(—)	(40)	(—)	(152)
C12200	H55	0.065	15	TYP	68	40	32	25	35	—	77	45	26
		(1.65)			(20)	(276)	(221)	(25)	(35)	(—)	(77)	(45)	(179)
	H80	0.065	40	TYP	68	55	50	8	60	—	95	63	29
		(1.65)			(20)	(379)	(345)	(8)	(60)	(—)	(95)	(63)	(200)
	OS025	0.065	0	TYP	68	34	11	45	—	—	45	—	23
		(1.65)			(20)	(234)	(76)	(45)	(—)	(—)	(45)	(—)	(159)
	OS050	0.065	0	TYP	68	32	10	45	—	—	40	—	22
		(1.65)			(20)	(221)	(69)	(45)	(—)	(—)	(40)	(—)	(152)
C14500	H55	0.065	15	TYP	68	40	32	20	35	—	—	—	24
		(1.65)			(20)	(276)	(221)	(20)	(35)	(—)	(—)	(—)	(165)
	OS050	0.065	0	TYP	68	32	10	40	—	—	40	—	22
		(1.65)			(20)	(221)	(69)	(40)	(—)	(—)	(40)	(—)	(152)
C18200	O61	—	0	TYP	68	40	15	50	—	—	59	—	—
C18400		(—)			(20)	(276)	(103)	(50)	(—)	(—)	(59)	(—)	(—)
	TD00	—	76	TYP	68	69	63	26	84	—	—	—	—
		(—)			(20)	(476)	(434)	(26)	(84)	(—)	(—)	(—)	(—)
	TH01	—	76	TYP	68	59	57	21	67	—	—	—	—
		(—)			(20)	(407)	(393)	(21)	(67)	(—)	(—)	(—)	(—)
C23000	H04	0.065	15	TYP	68	50	40	30	55	—	—	54	—
		(1.65)			(20)	(345)	(276)	(30)	(55)	(—)	(—)	(54)	(—)
	H04	0.065	35	TYP	68	70	58	8	77	—	—	68	—
		(1.65)			(20)	(483)	(400)	(8)	(77)	(—)	(—)	(68)	(—)
	OS015	0.065	0	TYP	68	44	18	45	—	—	71	38	—
		(1.65)			(20)	(303)	(124)	(45)	(—)	(—)	(71)	(38)	(—)
	OS050	0.065	0	TYP	68	40	12	55	—	—	60	15	—
		(1.65)			(20)	(276)	(83)	(55)	(—)	(—)	(60)	(15)	(—)

Table 6 (*Continued*)

UNS Alloy	Temper	Section Size in. (mm)	Cold Work %	Typ/ Min	Temp °F (°C)	Tensile Strength ksi (MPa)	Yield Strength (0.5% ext. under load) ksi (MPa)	El %	Rockwell Hardness				Shear Strength ksi (MPa)
									B	C	F	30T	
C26000	H04	— (—)	35	TYP	68 (20)	78 (538)	64 (441)	8 (8)	82 (82)	— (—)	— (—)	73 (73)	— (—)
	OS025	— (—)	0	TYP	68 (20)	52 (359)	20 (138)	55 (55)	— (—)	— (—)	75 (75)	40 (40)	— (—)
	OS050	— (—)	0	TYP	68 (20)	47 (324)	15 (103)	65 (65)	— (—)	— (—)	64 (64)	26 (26)	— (—)
C28000	H04	— (—)	30	TYP	68 (20)	74 (510)	55 (379)	10 (10)	80 (80)	— (—)	— (—)	— (—)	— (—)
	O50	— (—)	0	TYP	68 (20)	56 (386)	23 (159)	50 (50)	— (—)	— (—)	82 (82)	47 (47)	— (—)
C44300	OS025	— (—)	0	TYP	68 (20)	53 (365)	22 (152)	65 (65)	— (—)	— (—)	75 (75)	37 (37)	— (—)
C44400	OS025	— (—)	0	TYP	68 (20)	53 (365)	22 (152)	65 (65)	— (—)	— (—)	75 (75)	37 (37)	— (—)
C44500	OS025	— (—)	0	TYP	68 (20)	53 (365)	22 (152)	65 (65)	— (—)	— (—)	75 (75)	37 (37)	— (—)
C46400	H80	— (—)	35	TYP	68 (20)	88 (607)	66 (455)	18 (18)	95 (95)	— (—)	— (—)	— (—)	— (—)
C46500	H04	— (—)	35	TYP	68 (20)	88 (607)	66 (455)	18 (18)	95 (95)	— (—)	— (—)	— (—)	— (—)
C65500	H80	0.065 (1.7)	35	TYP	68 (20)	93 (641)	— (—)	22 (22)	92 (92)	— (—)	— (—)	78 (78)	— (—)
	OS050	0.065 (1.7)	0	TYP	68 (20)	57 (393)	— (—)	70 (70)	45 (45)	— (—)	— (—)	— (—)	— (—)
C68700	OS025	— (—)	0	TYP	68 (20)	60 (414)	27 (186)	55 (55)	— (—)	— (—)	77 (77)	— (—)	— (—)
C70600	H55	— (—)	0	TYP	68 (20)	60 (414)	57 (393)	10 (10)	72 (72)	— (—)	100 (100)	70 (70)	— (—)
	OS025	— (—)	0	TYP	68 (20)	44 (303)	16 (110)	42 (42)	15 (15)	— (—)	65 (65)	26 (26)	— (—)
C71500	OS025	— (—)	0	TYP	68 (20)	60 (414)	25 (172)	45 (45)	45 (45)	— (—)	80 (80)	— (—)	— (—)
	OS035	— (—)	0	TYP	68 (20)	54 (372)	— (—)	45 (45)	36 (36)	— (—)	77 (77)	— (—)	— (—)

is available in the annealed state (generally in coils) and in drawn tempers (straight lengths).

7.2 Commercial Tube and Fittings

Commercial tube is primarily used in air-conditioning and refrigeration systems. The tube is chemically similar to plumbing tube; however, dimensions are based on outside diameters, and some tubes are produced with enhanced inside and outside profiles for better heat transfer.

Other applications for copper tube include condenser, evaporator, and heat exchanger tubes; gas, heater, and oil burner lines; fire sprinkler systems; plumbing pipe and steam tubes; brewery and distillery tubes; gasoline, hydraulic, and oil lines, and rotating bands. A new designation, type G, refers to tube for fuel gas [both natural and liquid propane (LP) gas] distribution systems, a relatively new but rapidly growing application.

7.3 Alloy Tube

Copper–iron (UNS C19200), and in recent years, copper–nickel (UNS C70600) is specified for automotive hydraulic brake lines. The copper–nickel is especially resistant to stress–corrosion cracking and to attack by road salts.

Selection of alloys for condenser and heat exchanger tube is based on heat transfer rate (conductivity), corrosion resistance, and resistance to cavitation and biofouling. In general, conductivity is highest in pure copper and high-copper alloys, whereas mechanical and chemical properties are highest in more highly alloyed grades. Corrosion resistance of alloys cannot be ranked quantitatively since performance depends strongly on specific exposure conditions; however, resistance also generally increases with the degree of alloying.

Thus, the designer's choice ranges from simple brasses such as UNS C23000, tin brasses such as UNS C43500, UNS C44300, and UNS C68700 (arsenical aluminum brass), through silicon bronzes C65100 and C65500, aluminum bronzes such as UNS C60800, and finally copper–nickels UNS C70600 and C71500. Alloy C70600 is widely used for seacoast power plant condensers and critical piping systems on offshore platforms. The alloy's high biofouling resistance keeps piping systems free from marine growth. Alloy C71500, with 30% nickel, is used in severe-duty marine condensers and saltwater systems, as well as desalination equipment. Further information on copper tube, pipe and fittings, and on the selection of copper alloys and other commercial tube products can be found at http://piping.copper.org/ and http://www.copper.org/mechanical/homepage.htm, respectively.

8 ROD, BAR, AND MECHANICAL WIRE

Copper metals used in products manufactured by machining, hot forging, and cold heading are supplied as extruded and/or drawn rods, bars, and mechanical wire. When referring to copper products, *rod* means round, hexagonal, or octagonal forms supplied in straight lengths, i.e., not coiled. *Bar* products have square or rectangular cross sections and are also sold in straight lengths. *Shapes* are straight lengths with oval, half-round, geometric, or custom-ordered cross sections, and *wire* can have any cross section and is supplied in coils or on spools.

Products are specified by grade and temper. Temper definitions for wrought products are listed in Table 1. Mechanical properties corresponding to the most commonly selected tempers are listed in Table 5. The data are identical to values published in the *Society of Automotive Engineers (SAE) Handbook, Vol. 1, Materials,* Warrendale, PA (published annually).

8.1 Machined Products

By far, the alloy most often selected for machined products is UNS C36000, free-cutting brass. The alloy, a yellow brass containing a few percent of lead, is so readily machinable that it is accepted as the standard against which the machinability ratings of other metals are compared. Evaluations conducted in accordance with an established ASTM test method show that in the half-hard temper (H02), C36000 can theoretically be machined five times faster than SAE 12L14 free-machining steel at equivalent tool wear rates. Few automatic screw machines or machining centers can operate at such speeds, but the superior machinability of C36000 is also an important economic factor at conventional cutting rates. As a general rule of thumb, products in which more than about 55% of the starting rod is removed by machining—as is usually the case—will be less costly when made from C36000 compared with identical products made from 12L14.

When machined, free-cutting brass produces what are known as type I chips, small fragments that are easily removed from the cutting area. The chips form as a result of the alloy's lead content, which also acts as an internal lubricant and coolant. Other leaded copper alloys share this property to varying degrees. Type I alloys exhibit machinability ratings ranging from 100 (highest, for C36000) to 50. Type II chips are short, curly or serrated depending on alloy and the type of machining operation employed. Alloys displaying this form of chip have machinability ratings ranging from 60 to 20. While not quite as machinable as type I alloys, these grades offer higher mechanical properties and enhanced corrosion resistance, among other attributes. Alloys that form type III chips, which are long and tangled, are less well suited to high-speed automatic machining operations, and they are generally selected for other reasons. Machinability ratings range from 40 to 20, indicating that they can certainly be machined, only not at the high speeds available with leaded alloys. Therefore, when cost is a primary design consideration and production quantities are large enough to warrant the use of automatic screw machines or numerically controlled machining centers, free-cutting brass is normally the best starting point. When higher mechanical properties, specific corrosion resistance, or other special properties are needed, other copper alloys can be considered.

Brass chips are always recycled, and the sale of these chips partly offsets the metal's higher initial cost. Steel chips are difficult to recycle and have almost no value. Further savings are realized by the fact that brass products usually do not require plating or synthetic coatings for corrosion protection. Protective coatings add considerable cost and can give rise to environmental concerns, but brass's inherent corrosion resistance makes them unnecessary.

The yield strength of C36000 in the half-hard temper is 43 ksi (310 MPa). This value is somewhat lower than the strength of 12L14, but it is adequate for most screw-machined products. While the lead present in C36000 gives the metal its high machinability, it does restrict ductility somewhat. Products with severe knurling or thread-rolling in addition to machining can be specified in brasses such as C34500 and C35300, which contain less lead but have machinability ratings that are nearly as high as that of C36000.

Free-cutting brass (and other alloys) is also produced in custom-designed extruded shapes. Use of such shapes can cut machining time and reduce the number of components in an assembly. Custom extrusion dies are inexpensive and can normally be ready for use within a few days. A series of informative articles describing the properties and benefits of using free-cutting brass can be found at http://brassbar.copper.org/alloy360/homepage.ihtml. Additional literature on the material is available from CDA in print and on CD-ROM.

8.2 Forgings

Forging, or hot stamping, yields products with high, uniform mechanical properties and fine surface finishes. The process is often selected for products such as pump impellers, decorative architectural hardware and valves, and other pressure-retaining products where integrity is important. Because the forging process yields products with near net shapes, it competes with casting—especially permanent-mold and die casting—when large production runs are planned. Design limitations associated with forgings include avoidance of re-entrant angles and very thin sections and provision of relief angles of about 5° on axially oriented surfaces. On the other hand, forgings' dense structure, internal cleanliness, and consistent dimensions enable components to be designed with thinner walls than castings having equivalent pressure ratings. Most copper-base forgings are modest in size, although the process known as ring forging is capable of producing circular products as large as 25 ft (76 m) in diameter.

Many copper alloys, including brasses, nickel silvers, aluminum and silicon bronzes, copper–nickels, high-copper alloys, and copper itself are routinely forged. Forging brass, UNS C37700, is particularly well adapted to the process, offering die life up to 50,000 parts. It is a yellow brass, similar to free-cutting brass but containing only about one-half as much lead. The alloy shares many of C36000's properties, including good machinability (rating = 80) and corrosion resistance. Like other brasses, it can be polished to a high luster and readily accepts electroplating. Further information about copper-base forging alloys can be found at http://brassbar.copper.org/forgings.htm. Mechanical properties of forged copper alloys are listed along with rod, bar, and mechanical wire alloys in Table 5.

8.3 Mechanical Wire

Pure copper and single-phase copper alloys such as low-zinc brasses, copper–nickels, and high-copper alloys have exceptional ductility at low temperatures. This makes them good candidates for cost-effective forming processes such as cold heading. Countless screws, rivets, electrical connector pins, jewelry items, shafts, actuating arms, and high-strength, corrosion-resistant bolts are produced by way of such processes.

Cold working increases the strength and hardness of these alloys, adding to whatever other strengthening mechanisms may be present in the alloy (see Table 5). Cold working does not seriously inhibit an alloy's useful properties, although ductility obviously decreases, and susceptibility to stress corrosion cracking may increase in susceptible alloys and aggressive environments due to the presence of residual stresses. Electrical conductivity of cold-worked alloys will be slightly lower than that seen in annealed materials.

9 CASTINGS

Cast copper alloys are among the oldest fabricated materials. Ancient artisans only had copper and crude tin bronzes to work with, but today's designers continue to find new uses for cast copper alloys of all types. Cast copper alloys are specified for their favorable mechanical properties, good friction and wear properties, high conductivity, excellent machinability and fabricability, biofouling resistance, low manufactured cost, and attractive appearance. Mechanical property data for cast copper alloys are listed in Table 7.

Casting is often chosen over other manufacturing methods because it offers low cost. Copper alloys are not the lowest-cost raw materials, but they compete successfully with other cast metals—especially stainless steels and nickel-base alloys—because their predictable castability increases foundry yields, reducing rejection rates and keeping overall foundry costs low. The alloys' high machinability reduces the cost of secondary operations and enables the use of high-speed automatic machine tools that may not be suitable for other materials. Cast copper alloys are fully recyclable at the end of their service life and their metal value can eventually be recovered.

There are cast versions of many of the wrought alloys, and with certain minor exceptions the properties of alloys in corresponding families, whether wrought or cast, are also generally similar.

Temper designations for cast copper alloys are listed in Table 2. Most alloys are used in the as-manufactured (as-cast) or cast-and-annealed condition, and a few can be given postcasting heat treatments to enhance mechanical or other properties.

9.1 Casting Methods

Selection of copper alloys for cast products should be based on the product's technical requirements. The casting's size, shape, and complexity must also be considered. The casting method can also influence alloy selection. In general, copper metals can be cast using all conventional foundry methods: sand, permanent mold, continuous, centrifugal, investment, and plaster mold. Recent advances in the pressure die-casting method, which until now has almost exclusively been restricted to low-melting-point metals, will even enable this high-rate process to be applied to copper.

The metallurgical characteristics of individual copper alloys and, sometimes, entire alloy families make them more suitable for one or more casting methods and less well suited to others. The subject is too complex to be covered adequately here, but excellent relevant publications are available from the American Foundrymen's Society (AFS), the Non-Ferrous Founders Society (NFFS), and CDA. Useful information can also be gained by discussing product requirements with an experienced foundryman.

Briefly, the way an alloy solidifies largely determines its suitability to a given process or a particular product configuration. Metals that freeze at a fixed tem-

Table 7 Mechanical Properties of Selected Cast Coppers and Copper Alloys (Copper Development Association, Inc.)

UNS Alloy US (SI)	Method	Section Size in. (mm)	Typ/Spec Min	Temp °F (°C)	Tensile Strength ksi (MPa)	Yield Strength (0.5% ext. under load) ksi (MPa)	Yield Strength (0.2% offset) ksi (MPa)	El %	Brinell Hard. 500 kg	Brinell Hard. 3000 kg	Shear Strength ksi (MPa)	Fatigue Strength* ksi (MPa)	Izod Impact Strength ksi (MPa)
C80200 C81100	Sand	— (—)	TYP	68 (20)	25 (172)	9 (62)	— (—)	80 (80)	88 (88)	— (—)	— (—)	9 (62)	— (—)
C83450	Sand	— (—)	SMIN	68 (20)	35 (239)	18 (122)	18 (122)	50 (25)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	TYP	68 (20)	37 (255)	15 (103)	— (—)	31 (31)	62 (20)	— (—)	— (—)	— (—)	— (—)
C83600	Centr.	— (—)	SMIN	68 (20)	30 (205)	14 (97)	— (—)	20 (20)	— (—)	— (—)	— (—)	— (—)	— (—)
	Cont.	— (—)	SMIN	68 (20)	50 (345)	25 (170)	— (—)	12 (12)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	SMIN	68 (20)	30 (207)	14 (97)	— (—)	20 (20)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	TYP	68 (20)	35 (241)	17 (117)	— (—)	30 (30)	60 (60)	— (—)	— (—)	11 (76)	10 (14)
C84400	Centr.	— (—)	SMIN	68 (20)	29 (200)	13 (90)	— (—)	18 (18)	— (—)	— (—)	— (—)	— (—)	— (—)
	Cont.	— (—)	SMIN	68 (20)	30 (207)	15 (103)	— (—)	16 (16)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	SMIN	68 (20)	28 (193)	12 (83)	— (—)	16 (16)	55 (55)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	TYP	68 (20)	37 (255)	12 (83)	— (—)	35 (35)	55 (55)	— (—)	— (—)	— (—)	8 (11)
C84800	Centr.	— (—)	SMIN	68 (20)	29 (200)	13 (90)	— (—)	18 (18)	55 (55)	— (—)	— (—)	— (—)	— (—)
	Cont.	— (—)	SMIN	68 (20)	30 (207)	15 (103)	— (—)	16 (16)	55 (55)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	SMIN	68 (20)	28 (193)	12 (83)	— (—)	16 (16)	55 (55)	— (—)	— (—)	— (—)	
	Sand	— (—)	TYP	68 (20)	37 (255)	14 (97)	— (97)	35 (35)	55 (55)	— (—)	— (—)	— (—)	(—)

Table 7 (*Continued*)

UNS Alloy US (SI)	Method	Section Size in. (mm)	Typ/Spec Min	Temp °F (°C)	Tensile Strength ksi (MPa)	Yield Strength (0.5% ext. under load) ksi (MPa)	Yield Strength (0.2% offset) ksi (MPa)	El %	Brinell Hard. 500 kg	Brinell Hard. 3000 kg	Shear Strength ksi (MPa)	Fatigue Strength* ksi (MPa)	Izod Impact Strength ksi (MPa)
C85200	Centr.	— (—)	SMIN	68 (20)	35 (241)	12 (83)	— (—)	25 (25)	45 (45)	— (—)	— (—)	—	—
C85200	Centr.	— (—)	TYP	68 (20)	38 (262)	13 (90)	— (—)	35 (35)	45 (45)	— (—)	— (—)	—	—
	Sand	— (—)	TYP	68 (20)	35 (241)	12 (83)	— (—)	35 (35)	45 (45)	— (—)	— (—)	—	—
C85400	Centr.	— (—)	SMIN	68 (20)	30 (207)	11 (76)	— (—)	20 (20)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	SMIN	68 (20)	30 (207)	11 (76)	— (—)	20 (20)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	TYP	68 (20)	34 (234)	12 (83)	— (—)	35 (35)	50 (50)	— (—)	— (—)	— (—)	— (—)
C85700	Centr.	— (—)	SMIN	68 (20)	40 (276)	14 (97)	— (—)	15 (15)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	SMIN	68 (20)	40 (276)	14 (97)	— (—)	15 (15)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	TYP	68 (20)	50 (345)	18 (124)	— (—)	40 (40)	75 (75)	— (—)	— (—)	— (—)	— (—)
C86200	Centr.	— (—)	SMIN	68 (20)	90 (621)	— (—)	45 (310)	18 (18)	— (—)	— (—)	— (—)	— (—)	— (—)
	Cont.	— (—)	SMIN	68 (20)	90 (621)	— (—)	45 (310)	18 (18)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	SMIN	68 (20)	90 (621)	— (—)	45 (310)	18 (18)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	TYP	68 (20)	95 (655)	48 (331)	48 (331)	20 (20)	— (—)	180 (180)	— (—)	— (—)	12 (16)
C86300	Centr.	— (—)	SMIN	68 (20)	110 (758)	— (—)	60 (414)	12 (12)	— (—)	— (—)	— (—)	— (—)	— (—)
	Cont.	— (—)	SMIN	68 (20)	110 (758)	62 (427)	62 (427)	14 (14)	— (—)	— (—)	— (—)	— (—)	— (—)

	Sand	—	SMIN	68	110	—	60	12	—	—	—	—	—
		(—)		(20)	(768)	(—)	(414)	(12)	(—)	(—)	(—)	(—)	(—)
	Sand	—	TYP	68	119	—	67	—	—	225	—	25	15
		(—)		(20)	(821)	(—)	(462)	(—)	(—)	(225)	(—)	(172)	(20)
C86400	Centr.	—	SMIN	68	60	20	—	15	—	—	—	—	—
		(—)		(20)	(414)	(138)	(—)	(15)	(—)	(—)	(—)	(—)	(—)
C86400	Sand	—	SMIN	68	60	20	—	15	—	—	—	—	—
		(—)		(20)	(414)	(138)	(—)	(15)	(—)	(—)	(—)	(—)	(—)
	Sand	—	TYP	68	65	—	25	20	90	105	—	—	30
		(—)		(20)	(448)	(—)	(172)	(20)	(90)	(105)	(—)	(—)	(41)
C87300	Centr.	—	SMIN	68	45	18	—	20	85	—	—	—	—
		(—)		(20)	(310)	(124)	(—)	(20)	(—)	(—)	(—)	(—)	(—)
	Sand	—	SMIN	68	45	18	—	20	85	—	—	—	—
		(—)		(20)	(310)	(124)	(—)	(20)	(—)	(—)	(—)	(—)	(—)
C87500	Centr.	—	SMIN	68	60	24	—	16	—	—	—	—	—
		(—)		(20)	(414)	(165)	(—)	(16)	(—)	(—)	(—)	(—)	(—)
	PM	—	SMIN	68	80	30	—	15	—	—	—	—	—
		(—)		(20)	(552)	(207)	(—)	(15)	(—)	(—)	(—)	(—)	(—)
	Sand	—	TYP	68	67	30	—	21	115	134	—	22	—
		(—)		(20)	(462)	(207)	(—)	(21)	(115)	(134)	(—)	(152)	(—)
C89510	Sand	—	TYP	68	30	20	—	12	37	—	—	—	—
		(—)		(20)	(210)	(135)	(—)	(20)	(37)	(—)	(—)	(—)	(—)
C89550	Sand	—	MIN	68	35	21	20	5	—	—	—	—	—
		(—)		(20)	(240)	(140)	(135)	(5)	(—)	(—)	(—)	(—)	(—)
	Sand	—	TYP	68	48	29	28	8	—	—	—	—	—
		—		20	330	200	190	8	—	—	—	—	—
C90300	Cont.	—	SMIN	68	44	—	—	36	—	—	—	—	—
		(—)		(20)	(303)	(—)	(—)	(36)	(—)	(—)	(—)	(—)	(—)
	Sand		SMIN	68	40	18	—	30	70	—	—	—	
				(20)	(276)	(124)	(—)	(30)	(70)	(—)	(—)	(—)	

Table 7 (Continued)

UNS Alloy US (SI)	Method	Section Size in. (mm)	Typ/Spec Min	Temp °F (°C)	Tensile Strength ksi (MPa)	Yield Strength (0.5% ext. under load) ksi (MPa)	Yield Strength (0.2% offset) ksi (MPa)	El %	Brinell Hard.		Shear Strength ksi (MPa)	Fatigue Strength* ksi (MPa)	Izod Impact Strength ksi (MPa)
									500 kg	3000 kg			
	Sand		TYP	68	45	21	—	30	—	—	—	—	
				(20)	(310)	(145)	(—)	(30)	(—)	(—)	(—)	(—)	
C92200	Centr.	—	SMIN	68	34	16	—	24	65	—	—	—	11
		(—)		(20)	(234)	(110)	(—)	(24)	(65)	(—)	(—)	(—)	(76)
	Cont.	—	SMIN	68	28	19	—	18	—	—	—	—	—
		(—)		(20)	(262)	(131)	(—)	(18)	(—)	(—)	(—)	(—)	(—)
	PM	—	SMIN	68	68	32	—	24	—	—	—	—	—
		(—)		(20)	(468)	(220)	(—)	(24)	(—)	(—)	(—)	(—)	(—)
	Sand	—	SMIN	68	34	16	—	24	65	—	—	—	11
		(—)		(20)	(234)	(110)	(—)	(24)	(65)	(—)	(—)	(—)	(76)
C92200	Sand	—	TYP	68	40	20	—	30	65	—	—	—	12
		—		20	276	138	—	30	65	—	—	—	76
C92300	Centr.	—	SMIN	68	36	16	—	18	70	—	—	—	—
		(—)		(20)	(248)	(110)	(—)	(18)	(70)	(—)	(—)	(—)	(12)
	Cont.	—	SMIN	68	80	38	—	32	—	—	—	—	76
		(—)		(20)	(552)	(262)	(—)	(32)	(—)	(—)	(—)	(—)	(—)
	Sand	—	SMIN	68	36	16	—	18	—	—	—	—	—
		(—)		(20)	(248)	(110)	(—)	(18)	(—)	(—)	(—)	(—)	(—)
	Sand	—	TYP	68	40	20	—	25	70	—	—	—	12
		(—)		(20)	(276)	(138)	(—)	(25)	(70)	(—)	(—)	(—)	(76)
C92600	Sand	—	SMIN	68	40	18	—	20	—	—	—	—	—
		(—)		(20)	(276)	(124)	(—)	(20)	(—)	(—)	(—)	(—)	(—)
	Sand	—	TYP	68	44	20	—	30	70	—	—	—	7
		(—)		(20)	(303)	(138)	(—)	(30)	(70)	(—)	(—)	(—)	(9)
C93200	Centr.	—	SMIN	68	30	14	—	15	—	—	—	—	—
		(—)		(20)	(207)	(97)	(—)	(15)	(—)	(—)	(—)	(—)	(—)
	Cont.	—	SMIN	68	35	20	—	10	—	—	—	—	—
		(—)		(20)	(241)	(138)	(—)	(10)	(—)	(—)	(—)	(—)	(—)
	Sand	—	SMIN	68	30	14	—	15	—	—	—	—	—
		(—)		(20)	(207)	(97)	(—)	(15)	(—)	(—)	(—)	(—)	(—)

	Sand	— (—)	TYP	68 (20)	35 (241)	18 (124)	— (—)	20 (20)	65 (65)	— (—)	— (—)	16 (110)	6 (8)
C93500	Centr.	— (—)	SMIN	68 (20)	28 (193)	12 (83)	— (—)	15 (15)	— (—)	— (—)	— (—)	— (—)	— (—)
	Cont.	— (—)	SMIN	68 (20)	60 (412)	32 (220)	— (—)	24 (24)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	SMIN	68 (20)	28 (193)	12 (83)	— (—)	15 (15)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	TYP	68 (20)	32 (221)	18 (224)	— (—)	12 (12)	60 (60)	— (—)	— (—)	— (—)	— (—)
C93700	Centr.	— (—)	SMIN	68 (20)	60 (412)	24 (168)	— (—)	30 (30)	60 (60)	— (—)	18 (124)	— (—)	5 (7)
	Cont.	— (—)	SMIN	68 (20)	35 (241)	20 (128)	— (—)	6 (6)	— (—)	— (—)	— (—)	— (—)	— (—)
C93700	Sand	— (—)	SMIN	68 (20)	30 (207)	12 (283)	— (—)	15 (15)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	TYP	68 (20)	35 (241)	18 (124)	16 (110)	20 (20)	60 (60)	— (—)	18 (124)	13 (90)	5 (7)
C93800	Centr.	— (—)	SMIN	68 (20)	26 (179)	14 (197)	— (—)	12 (12)	— (—)	— (—)	— (—)	— (—)	— (—)
	Centr.	— (—)	TYP	68 (20)	30 (207)	16 (110)	— (—)	18 (18)	— (—)	— (—)	— (—)	— (—)	— (—)
	Cont.	— (—)	SMIN	68 (20)	25 (172)	16 (110)	— (—)	5 (5)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	SMIN	68 (20)	26 (179)	14 (197)	— (—)	12 (12)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	TYP	68 (20)	30 (207)	16 (110)	— (—)	18 (18)	55 (55)	— (—)	15 (103)	10 (69)	5 (7)
C94300	Centr.	— (—)	SMIN	68 (20)	21 (145)	30 (103)	— (—)	10 (10)	— (—)	— (—)	— (—)	— (—)	— (—)

Table 7 (*Continued*)

UNS Alloy US (SI)	Method	Section Size in. (mm)	Typ/Spec Min	Temp °F (°C)	Tensile Strength ksi (MPa)	Yield Strength (0.5% ext. under load) ksi (MPa)	Yield Strength (0.2% offset) ksi (MPa)	El %	Brinell Hard.		Shear Strength ksi (MPa)	Fatigue Strength* ksi (MPa)	Izod Impact Strength ksi (MPa)
									500 kg	3000 kg			
	Cont.	— (—)	SMIN	68 (20)	21 (145)	15 (103)	— (—)	7 (7)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	SMIN	68 (20)	21 (145)	— (—)	— (—)	10 (10)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	TYP	68 (20)	27 (186)	13 (90)	— (—)	15 (15)	48 (48)	— (—)	— (—)	— (—)	5 (7)
C95200	Centr.	— (—)	SMIN	68 (20)	65 (448)	25 (172)	— (—)	20 (20)	— (—)	— (—)	— (—)	— (—)	— (—)
	Cont.	— (—)	SMIN	68 (20)	68 (469)	26 (169)	— (—)	20 (20)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	SMIN	68 (20)	65 (448)	25 (172)	— (—)	20 (20)	— (—)	— (—)	— (—)	— (—)	— (—)
C95300	Centr.	— (—)	SMIN	68 (20)	65 (448)	25 (186)	— (—)	20 (20)	— (—)	140 (140)	41 (283)	22 (152)	28 (38)
	TQ50	— (—)	SMIN	68 (20)	80 (552)	85 (586)	— (—)	12 (12)	— (—)	174 (174)	46 (317)	27 (186)	— (—)
	Cont.	— (—)	SMIN	68 (20)	70 (483)	26 (179)	— (—)	25 (25)	— (—)	— (—)	— (—)	— (—)	— (—)
C95300	PM	— (—)	SMIN	68 (20)	80 (550)	30 (205)	— (—)	20 (20)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	SMIN	68 (20)	65 (448)	25 (172)	— (—)	20 (20)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	TYP	68 (20)	75 (517)	27 (186)	— (—)	25 (25)	— (—)	140 (140)	41 (283)	22 (152)	28 (38)
	TQ50	— (—)	SMIN	68 (20)	80 (552)	40 (276)	— (—)	12 (12)	— (—)	— (—)	— (—)	— (—)	— (—)
	TQ50	— (—)	TYP	68 (20)	85 (586)	42 (290)	— (—)	15 (15)	— (—)	174 (174)	46 (317)	27 (186)	24 (33)
C95400	Centr.	— (—)	SMIN	68 (20)	75 (517)	30 (207)	— (—)	12 (12)	— (—)	— (—)	— (—)	— (—)	— (—)

	TQ50	— (—)	SMIN	68 (20)	90 (621)	45 (310)	— (—)	12 (12)	— (—)	— (—)	— (—)	— (—)	— (—)
	Cont.	— (—)	SMIN	68 (20)	85 (586)	32 (221)	— (—)	12 (12)	— (—)	— (—)	— (—)	— (—)	— (—)
	TQ50	— (—)	SMIN	68 (20)	95 (655)	45 (310)	— (—)	10 (10)	— (—)	— (—)	— (—)	— (—)	— (—)
	PM	— (—)	SMIN	68 (20)	100 (690)	40 (275)	— (—)	10 (10)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	SMIN	68 (20)	75 (517)	30 (207)	— (—)	12 (12)	— (—)	— (—)	— (—)	—	—
	Sand	— (—)	TYP	68 (20)	85 (586)	35 (241)	— (—)	18 (18)	— (—)	170 (170)	47 (324)	28 (193)	16 (22)
	TQ50	— (—)	SMIN	68 (20)	90 (621)	45 (310)	— (—)	6 (6)	— (—)	— (—)	— (—)	— (—)	— (—)
	TQ50	— (—)	TYP	68 (20)	105 (724)	54 (372)	— (—)	8 (8)	— (—)	195 (195)	— (—)	35 (241)	11 (15)
	Cast & Annealed	— (—)	SMIN	68 (20)	75 (517)	30 (207)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)	— (—)
C95500	Centr.	— (—)	SMIN	68 (20)	90 (621)	40 (276)	— (—)	6 (6)	— (—)	— (—)	— (—)	— (—)	— (—)
	TQ50	— (—)	SMIN	68 (20)	110 (758)	60 (414)	— (—)	5 (5)	— (—)	— (—)	— (—)	— (—)	— (—)
C95500	Cont.	— (—)	SMIN	68 (20)	95 (565)	42 (290)	— (—)	10 (10)	— (—)	— (—)	— (—)	— (—)	— (—)
	TQ50	— (—)	SMIN	68 (20)	110 (758)	60 (414)	— (—)	8 (8)	— (—)	— (—)	— (—)	— (—)	— (—)
	Centr.	— (—)	TYP	68 (20)	100 (690)	44 (303)	— (—)	12 (12)	— (—)	— (—)	— (—)	— (—)	— (—)
	PM	— (—)	SMIN	68 (20)	110 (760)	60 (415)	— (—)	5 (5)	— (—)	— (—)	— (—)	— (—)	— (—)

Table 7 (*Continued*)

UNS Alloy US (SI)	Method	Section Size in. (mm)	Typ/Spec Min	Temp °F (°C)	Tensile Strength ksi (MPa)	Yield Strength (0.5% ext. under load) ksi (MPa)	Yield Strength (0.2% offset) ksi (MPa)	El %	Brinell Hard.		Shear Strength ksi (MPa)	Fatigue Strength* ksi (MPa)	Izod Impact Strength ksi (MPa)
									500 kg	3000 kg			
	Sand	— (—)	SMIN	68 (20)	90 (621)	40 (276)	— (—)	6 (6)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	TYP	68 (20)	100 (690)	44 (303)	— (—)	12 (12)	— (—)	195 (195)	48 (331)	31 (214)	13 (18)
C95700	Cont.	— (—)	SMIN	68 (20)	90 (620)	40 (275)	— (—)	15 (15)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	SMIN	68 (20)	90 (620)	40 (275)	— (—)	20 (20)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	TYP	68 (20)	95 (655)	45 (310)	— (—)	26 (26)	— (—)	180 (180)	— (—)	33 (228)	20 (27)
C95800	Centr.	— (—)	SMIN	68 (20)	85 (586)	35 (241)	— (—)	15 (15)	— (—)	— (—)	— (—)	— (—)	— (—)
	Cont.	— (—)	SMIN	68 (20)	90 (621)	38 (262)	— (—)	18 (18)	— (—)	— (—)	— (—)	— (—)	— (—)
	PM	— (—)	SMIN	68 (20)	90 (620)	40 (275)	— (—)	15 (15)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	—	SMIN	68 (20)	85 (586)	35 (241)	— (—)	15 (15)	— (—)	— (—)	— (—)	— (—)	—
	Sand	— (—)	TYP	68 (20)	95 (655)	38 (262)	— (—)	25 (25)	— (—)	159 (159)	58 (400)	31 (214)	20 (27)
C96400	Sand	— (—)	SMIN	68 (20)	60 (415)	32 (220)	— (—)	20 (20)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	TYP	68 (20)	68 (469)	37 (255)	— (—)	28 (28)	— (—)	140 (140)	— (—)	18 (124)	— (—)
C97300	Centr.	— (—)	SMIN	68 (20)	30 (207)	15 (103)	— (—)	8 (8)	— (—)	— (—)	— (—)	— (—)	— (—)
	Cont.	— (—)	SMIN	68 (20)	30 (207)	15 (103)	— (—)	10 (10)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	SMIN	68 (20)	30 (241)	15 (103)	— (—)	8 (8)	— (—)	— (—)	— (—)	— (—)	— (—)

	Sand	— (—)	TYP	68 (20)	35 (241)	17 (117)	— (—)	20 (20)	55 (55)	— (—)	— (—)	— (—)	— (—)
C97400	Sand	— (—)	TYP	68 (20)	38 (262)	17 (117)	— (—)	20 (20)	70 (70)	— (—)	— (—)	— (—)	— (—)
C97600	Centr.	— (—)	SMIN	68 (20)	40 (276)	17 (117)	— (—)	10 (10)	— (—)	— (—)	— (—)	— (—)	— (—)
	Cont.	— (—)	SMIN	68 (20)	40 (276)	20 (138)	— (—)	10 (10)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	SMIN	68 (20)	120 (828)	51 (351)	— (—)	20 (20)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	TYP	68 (20)	45 (310)	24 (165)	— (—)	20 (20)	80 (80)	— (—)	— (—)	16 (107)	— (—)
C97800	Centr.	— (—)	SMIN	68 (20)	50 (345)	22 (152)	— (—)	10 (10)	— (—)	— (—)	— (—)	— (—)	— (—)
	Cont.	— (—)	SMIN	68 (20)	45 (310)	22 (152)	— (—)	8 (8)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	SMIN	68 (20)	50 (345)	22 (152)	— (—)	10 (10)	— (—)	— (—)	— (—)	— (—)	— (—)
	Sand	— (—)	TYP	68 (20)	55 (379)	30 (207)	— (—)	15 (15)	— (—)	130 (130)	— (—)	— (—)	— (—)

*Fatigue strength: 100 × 10^6 cycles unless indicated as [N] × 10^6.

perature (e.g., pure copper) or over a narrow temperature range (yellow brasses) tend to be more forgiving of solidification rate and can generally be cast by a variety of methods. Alloys with broad freezing ranges require slower solidification rates (as in sand casting) in order to avoid excessive internal porosity. These are not hard and fast rules, however, and they can often be abridged by careful design and foundry practice.

A better understanding of the importance of freezing behavior has led to a growing interest in the permanent-mold casting process in the United States. The process makes use of ‘‘permanent’’ metal dies that induce rapid solidification and therefore enable short cycle times and high production rates. Among the process’s other advantages are the ability to produce near net shapes, fine surface finishes, close tolerances, and exceptional part-to-part uniformity. Thin section sizes are also readily attainable. The permanent-mold process is considered friendly to the environment because it leaves virtually no residues for disposal. From the designer’s standpoint, the method’s most significant advantage is that the dense structure and fine grain size it produces results in castings having higher strength, for the same alloys, than those available in sand castings. For alloy C87500, a silicon brass, tensile strengths (TS) for sand-cast and permanent-mold cast versions are 462 MPa (67 ksi) and 562 MPs (80 ksi), respectively, a 21% difference. Table 7 contains several other examples that illustrate this phenomenon.

9.2 Uses

The plumbing industry is the largest user of copper castings, mainly for brass fixtures, fittings, and water meters. Among the commonly used alloys are C83600, a leaded red brass, C84400, a semired brass, and several of the yellow brasses, the latter often being specified for decorative faucets and similar hardware.

Traditional plumbing brasses contain lead to improve castability and machinability. Concerns expressed in recent years over the possibility that a portion of the lead might be leached from a plumbing fixture’s internal surfaces by aggressive water and thus enter the human food chain led to the development of several new alloys, aptly named EnviroBrasses. These brasses contain only trace amounts of lead up to a maximum of 0.25%. EnviroBrass I (C89510) and EnviroBrass II (C89520) substitute a mixture of selenium and bismuth for the lead contained in conventional red and semired brasses. EnviroBrass III (C89550) is a yellow brass that is ideally suited to the permanent-mold casting process.

Industrial pumps, valves, and fittings are other important outlets for copper alloy castings. Alloys are generally selected for favorable combinations of corrosion, wear, and mechanical properties. Popular alloys include aluminum bronze, nickel–aluminum bronze, tin bronzes, manganese bronze, and silicon bronzes and brasses.

Copper alloys have been used in marine products for centuries, and that trend continues today. The copper metals exhibit excellent general corrosion resistance in both fresh and seawater. Unlike some stainless steels, they resist pitting and stress-induced cracking in aggressive chloride environments.

Commonly selected alloys include dezincification-inhibited brasses, tin bronzes, and manganese, silicon, and aluminum bronzes. For maximum seawater corrosion resistance, copper–nickels should be considered.

Decorative architectural hardware is often cast in yellow brass. Plaques and statuary make use of the copper metals' ability to reproduce fine details, and the alloys' wide range of colors—including natural and synthetic patinas—have long been favored by artists and designers.

9.3 Sleeve Bearings

Sleeve bearings deserve mention here because, with the exception of oil-impregnated powder metal bearings, most bronze bearings are produced as either continuous or centrifugal castings. Design of sleeve bearings is based on design loads, operating speeds, temperature, and lubricant and lubrication mode. Selection of the optimum alloy for a particular design takes all these factors into account; however, journal hardness and alignment, possible lubricant starvation, and other unusual operating conditions must also be considered.

Tin bronzes, leaded tin bronzes, and high-leaded tin bronzes are the most commonly specified sleeve bearing alloys, alloy C93200 being considered the workhorse of the industry. Tin imparts strength; lead improves antifrictional properties but does so at the expense of some strength. High-leaded tin bronzes have the highest lubricity but the lowest strength of the bearing alloys. Aluminum bronzes and manganese bronzes are selected for applications that require very high strength and excellent corrosion resistance.

A useful primer on sleeve bearing design can be found at http://www.copper.org/industrial/bronze bearing.htm. PC-compatible sleeve bearing design software is available from CDA.

10 COPPER IN HUMAN HEALTH AND THE ENVIRONMENT

There has been a trend recently for engineers to take a material's health and environmental effects into account during product design, and "heavy metals" such as lead and cadmium, alone or in alloyed form, have lost favor despite whatever benefit they brought to market. While copper is chemically defined as a heavy metal, its use should give designers no concerns in this regard. Copper has, in fact, rightly been called an environmentally "green" metal.

Copper is essential to human, animal, and plant life. It is especially important to expectant mothers and infants. Without sufficient dietary intake to maintain internal stores, people suffer metabolic disorders and a variety of other problems. Animals fail to grow properly when copper is not provided in their feed or if they graze on copper-deficient plants. Crops grown on copper-deficient soils produce lower yields and some plants may simply wither and die.

On the other hand, copper does exhibit toxicity under some circumstances. This property is exploited beneficially in, for example, antifouling marine paints, agricultural fungicides, and alloys for seawater piping systems. In the United States, federal regulations limit public water supplies to copper concentrations to 1.3 parts per million (ppm)—the limit in Europe is 2.0 ppm—but higher levels found in some well waters are objectionable mainly because of the metallic taste the metal imparts. The threshold for acute physiological effects, mainly nausea and other temporary gastrointestinal disorders, is estimated to lie between 4 and 6 ppm, although sensitivity varies widely among individuals.

There has been concern expressed over the discharge of copper in effluents from copper roofs and copper plumbing systems. Copper may exist in such effluents in minute quantities, mainly bound as chemical compounds and complexes that are not ecologically available. Such forms of copper differ from ionic copper, which can exhibit ecotoxicity but which appears to exist only briefly in nature owing to copper's very strong bonding tendency, leading it to form non-bioavailable or nontoxic chemical compounds.

Finally, the fact that almost all copper is eventually recycled into useful products deserves recognition as one of the metal's environmental benefits. Currently, about 45% of all copper in use has been used in some form before. Largely because of the metal's high value, almost none of it finds its way to landfills. Recycling not only conserves a natural resource, it also avoids re-expenditure of the energy needed for mining, smelting and, refining.

CHAPTER 6

SELECTION OF TITANIUM ALLOYS FOR DESIGN

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1 INTRODUCTION

1.1 Purpose

The purpose of this chapter is to provide a reasonable understanding of titanium and its alloys so that selection of them for specific designs will be appropriate.

Handbook of Materials Selection. Edited by Myer Kutz

Knowledge of titanium alloy types and their processing should provide a potential user with a better ability to understand the ways in which titanium alloys (and titanium) can contribute to a design. Furthermore, the knowledge provided here should enable a user of titanium alloys to:

- Ask the important production questions of titanium providers.
- Specify necessary mechanical property levels to optimize alloy performance in the desired application.
- Determine the relevant corrosion/environmental factors that may affect a component.

Properties of the titanium alloy families sometimes are listed in handbooks or vendor/supplier brochures. However, not all data will be available and there is no special formula for titanium alloy selection.

Larger volume customers frequently dictate the resultant material conditions that generally will be available from a supplier. Proprietary alloy chemistries and/or proprietary/restricted processing required by customers can lead to alloys that may not be widely available. In general, proprietary processing is more likely to be encountered than proprietary chemistries nowadays. With few exceptions, critical applications for titanium and its alloys will require the customer to work with one or more titanium producers to develop an understanding of what is available and what a selector/designer can expect from a chosen titanium alloy.

1.2 What Are Titanium Alloys?

Titanium alloys for purposes of this chapter are those alloys of about 50% or higher titanium that offer exceptional strength-to-density benefits plus corrosion properties comparable to the excellent corrosion resistance of pure titanium. The range of operation is from cryogenic temperatures to around 538°C (1000°F) or slightly higher. Titanium alloys based on intermetallics such as gamma titanium aluminide (TiAl intermetallic compound that has been designated γ) are included in this discussion but offer no clear-cut mechanical advantages for now and an economic debit in many instances.

1.3 Temperature Capability of Titanium Alloys

The melting point of titanium is in excess of 1660°C (3000°F), but commercial alloys operate at substantially lower temperatures. It is not possible to create titanium alloys that operate close to their melting temperatures. Attainable strengths, crystallographic phase transformations, and environmental interaction considerations cause restrictions. Thus, while titanium and its alloys have melting points higher than those of steels, their maximum upper useful temperatures for structural applications generally range from as low as 427°C (800°F) to the region of about 538–595°C (1000–1100°F) dependent on composition. Titanium aluminide alloys show promise for applications at temperatures up to 760°C (1400°F).

Actual application temperatures will vary with individual alloy composition. Since application temperatures are much below the melting points, incipient melting is not a factor in titanium alloy application.

1.4 Strength and Corrosion Capability of Titanium and Its Alloys

Titanium owes its industrial use to two significant factors:

- Titanium has exceptional room temperature resistance to a variety of corrosive media.
- Titanium has a relatively low density and can be strengthened to achieve outstanding properties when compared with competitive materials on a strength to density basis.

Table 1 compares typical strength-to-density values for commercial purity (CP) titanium, several titanium alloys, and a high strength steel. Figure 1 depicts the strength improvements possible in titanium alloys. In addition to the excellent strength characteristics, titanium's corrosion resistance makes it a desirable material for body replacement parts and other tough corrosion-prone applications.

1.5 Titanium Alloy Information

While some chemistries and properties are listed in this chapter, there is no substitute for consultation with titanium manufacturers about the forms (some cast, mostly wrought) that can be provided and the exact chemistries available. It should be understood that not all titanium alloys, particularly those with specific processing, are readily available as off-the-shelf items. Design data for titanium alloys are not intended to be conveyed here, but typical properties are indicated for some materials. Design properties should be obtained from internal testing if possible, or from producers or other validated sources if sufficient test data are not available in-house. Typical properties are merely a guide for comparison. Exact chemistry, section size, heat treatment, and other processing steps must be known to generate adequate property values for design.

The properties of titanium alloy compositions that have been developed over the years are not normally well documented in the literature. However, since many consumers actually only use a few alloys within the customary user groups, data may be more plentiful for certain compositions. In the case of titanium, the most used and studied alloy, whether wrought or cast, is Ti–6Al–4V.

The extent to which data generated for specific applications are available to the general public is unknown. However, even if such data were disseminated widely, the alloy selector needs to be aware that processing operations such as forging conditions, heat treatment, etc. dramatically affect properties of titanium

Table 1 Comparison of Typical Strength-to-Weight Ratios at 20°C

Metal	Specific Gravity	Tensile Strength (lb/in.^{2})	Tensile Strength ÷ Specific Gravity
CP	4.5	58,000	13,000
Ti–6Al–4V	4.4	130,000	29,000
Ti–4Al–3Mo–1V	4.5	200,000	45,000
Ultrahigh-strength steel (4340)	7.9	287,000	36,000

Source: From *Titanium: A Technical Guide,* 1st ed., ASM International, Materials Park, OH 44073-0002, 1988, p. 158.

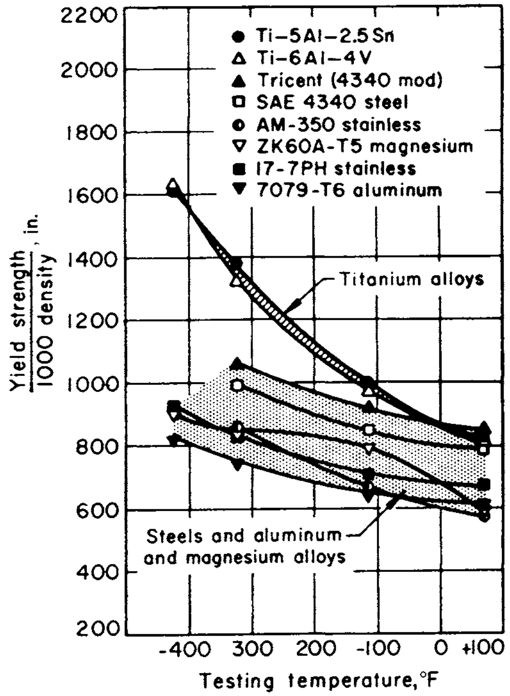


Fig. 1 Yield strength-to-density ratio as a function of temperature for several titanium alloys compared to some steel, aluminum, and magnesium alloys. (From *Titanium: A Technical Guide,* 1st ed., ASM International, Materials Park, OH 44073-0002, 1988, p. 158.)

alloys. All data should be reconciled with the actual manufacturing specifications and processing conditions expected. Alloy selectors should work with competent metallurgical engineers to establish the validity of data intended for design as well as to specify the processing conditions that will be used for component production.

Application of design data must take into consideration the probability of components containing locally inhomogeneous regions. For titanium alloys, such segregation can be disastrous in gas turbine applications. The probability of occurrence of these regions is dependent upon the melting procedures, being essentially eliminated by so-called triple melt. All facets of chemistry and processing need to be considered when selecting a titanium alloy for an application.

For sources of property data other than that of the producers (melters, forgers, etc.) or an alloy selector's own institution, one may refer to handbooks or organizations, such as ASM International, that publish compilations of data that may form a basis for the development of design allowables for titanium alloys. Standards organizations such as the American Society for Testing and Materials (ASTM) publish information about titanium alloys, but that information may not ordinarily contain any design data.

2 METALLURGY OF TITANIUM ALLOYS

2.1 Structures

Metals are crystalline and the atoms take various crystallographic forms. Some of these forms tend to be associated with better property characteristics than other crystal structures. Titanium, as does iron, exists in more than one crystallographic form. Titanium has two elemental crystal structures: in one, the atoms are arranged in a body-centered cubic array, in the other they are arranged in a close-packed hexagonal array. The cubic structure is found only at high temperatures, unless the titanium is alloyed with other elements to maintain the cubic structure at lower temperatures.

We are concerned not only with crystal structure but also overall "structure," i.e., appearance, at levels above that of atomic crystal structure. Structure for our purposes will be defined as the macrostructure and microstructure (i.e., macro- and microappearance) of a polished and etched cross section of metal visible at magnifications up to and including 10,000×. Two other microstructural features that are not determined visually but are determined by other means such as X-ray diffraction or chemistry are: phase type (e.g., α and β) and texture (orientation) of grains.

Titanium's two crystal structures are commonly known as α and β. Alpha actually means any hexagonal titanium, pure or alloyed, while beta means any cubic titanium, pure or alloyed. The α and β structures—sometimes called systems or types—are the basis for the generally accepted classes of titanium alloys. These are α, near-α, α–β, and β. Sometimes a category of near-β is also considered. The preceding categories denote the general type of microstructure after processing.

Crystal structure and grain structure (a component of microstructure) are not synonymous terms. Both (as well as the arrangement of phases in the microstructure) must be specified to completely identify the alloy and its expected mechanical, physical, and corrosion behavior. The important fact to keep in mind is that, while grain shape and size do affect behavior, the crystal structure changes (from α to β and back again) which occur during processing play a major role in defining titanium properties.

2.2 Crystal Structure Behavior in Alloys

An α-alloy (so described because its chemistry favors α-phase) does not normally form β-phase on heating. A near-α (sometimes called "superalpha") alloy forms only limited β-phase on heating, and so it may appear microstructurally similar to an α-alloy when viewed at lower temperatures. An α–β alloy is one for which the composition permits complete transformation to β on heating but transformation back to α plus retained and/or transformed β at lower temperatures. A near-β or β-alloy composition tends to retain, indefinitely at lower temperatures, the β-phase formed at high temperatures. However, the β that is retained on initial cooling to room temperature is metastable for many β-alloys. Dependent on chemistry, it may precipitate secondary phases during heat treatment.

3 METALS AT HIGH TEMPERATURES

3.1 General

While material strengths at low temperatures are usually not a function of time, at high temperatures the time of load application becomes very significant for mechanical properties. Concurrently, the availability of oxygen at high temperatures accelerates the conversion of some of the metal atoms to oxides. Oxidation proceeds much more rapidly at high temperatures than at room or lower temperatures. For alloys of titanium there is the additional complication of titanium's high affinity for oxygen and its ability to "getter" oxygen (or nitrogen) from the air. Dissolved oxygen greatly changes the strength and ductility of titanium alloys. Hydrogen is another gaseous element that can significantly affect properties of titanium alloys. Hydrogen tends to cause hydrogen embrittlement while oxygen (and nitrogen) will increase strength and reduce the ductility but not necessarily embrittle titanium as hydrogen does.

3.2 Mechanical Behavior

In the case of short-time tensile properties of yield strength (TYS) and ultimate strength (UTS), the mechanical behavior of metals at higher temperatures is similar to that at room temperature but with metals becoming weaker as the temperature increases. However, when steady loads below the normal yield or ultimate strength determined in short-time tests are applied for prolonged times at higher temperatures, the situation is different. Figure 2 illustrates the way in which most materials respond to steady extended-time loads at high temperatures. A time-dependent extension (creep) is noticed under load. If the alloy is exposed for a long time, the alloy eventually fractures (ruptures). The degradation process is called creep or, in the event of failure, creep-rupture (sometimes stress-rupture), and alloys are selected on their ability to resist creep and creep-rupture failure. Cyclically applied loads that cause failure (fatigue) at lower temperatures also cause failures in shorter times (lesser cycles) at high temperatures. When titanium alloys operate for prolonged times at high temperature, they can fail by creep-rupture. However, tensile strengths, fatigue strengths, and crack propagation criteria are more likely to dominate titanium alloy performance requirements.

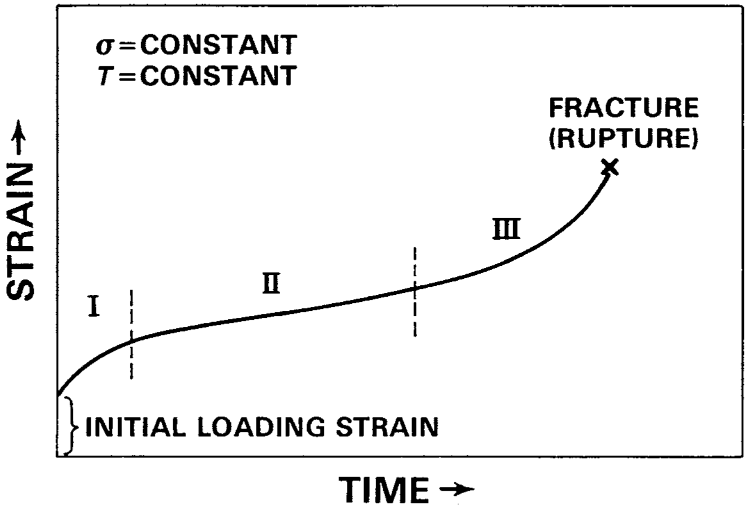
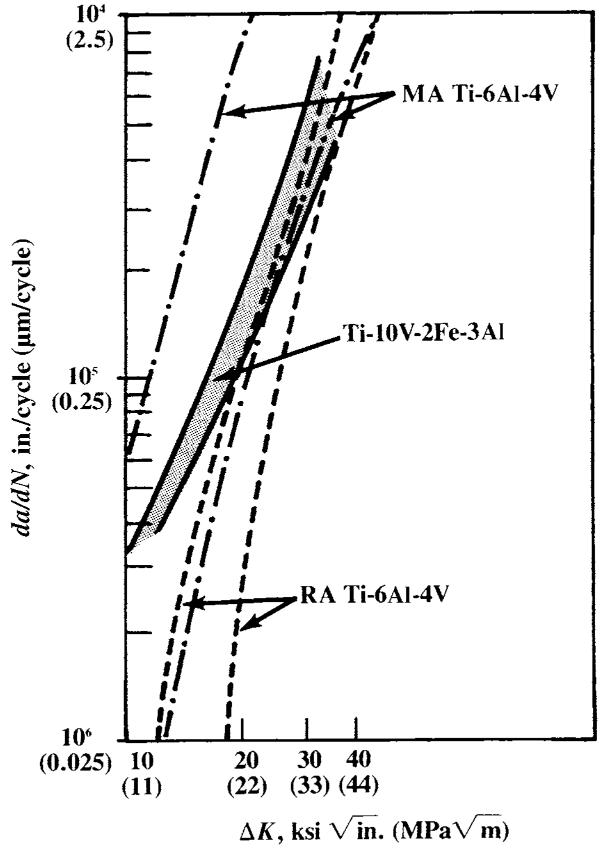


Fig. 2 Creep rupture schematic showing time-dependent deformation under constant load at constant high temperatures followed by final rupture. (All loads below the short time yield strength. Roman numerals denote ranges of the creep rupture curve.)

In highly mechanically loaded parts, such as gas turbine compressor disks, a common titanium alloy application, fatigue at high loads in short times, low cycle fatigue (LCF) is the major concern. High cycle fatigue (HCF) normally is not a problem with titanium alloys unless a design error occurs and subjects a component to a high-frequency vibration that forces rapid accumulation of fatigue cycles. While life under cyclic load (*S–N* behavior) is a common criterion for design, resistance to crack propagation is an increasingly desired property. Thus, the crack growth rate versus a fracture toughness parameter is required. The parameter in this instance is the stress intensity factor (K) range over an incremental distance which a crack has grown—the difference between the maximum and minimum K in the region of crack length measured. A plot of the resultant type (da/dn vs. ΔK) is shown in Fig. 3 for several wrought titanium alloys.



For Ti–10V–2Fe–3Al: $R = 0.05$; $F = 1$–30 Hz
For MA Ti–6Al–4V: $R = 0.08$; $F = 1$–25 Hz
For RA Ti–6Al–4V: $R = 0.08$; $F = 6$ Hz

Fig. 3 Comparison of fatigue crack growth rate (*da/dn*) vs. toughness change (Δ*K*). Curves for several titanium alloys. Note that MA = mill annealed while RA = recrystallization annealed. (From *Titanium: A Technical Guide,* 1st ed., ASM International, Materials Park, OH 44073-0002, 1988, p. 184.)

4 MICROSTRUCTURE AND PROPERTIES OF TITANIUM AND ITS ALLOYS

The grain size, grain shape, and grain boundary arrangements in titanium have a very significant influence on mechanical properties, and it is the ability to manipulate the phases/grains present as a result of alloy composition that is responsible for the variety of properties that can be produced in titanium and its alloys. Transformed β-phase products in alloys can affect tensile strengths, ductility, toughness, and cyclic properties. To these effects, must be added the basic strengthening effects of alloy elements.

4.1 Alloy Composition and General Behavior

Alpha alloys usually have high amounts of aluminum that contribute to oxidation resistance at high temperatures. (α–β alloys also contain, as the principal element, high amounts of aluminum, but the primary reason is to control the α-phase.) Alpha alloys cannot be heat treated to develop higher mechanical properties because they are single-phase alloys.

The addition of certain alloy elements to pure titanium provides for a wide two-phase α plus β region and so enables the resultant alloys to be heat treated or processed in the temperature range where the alloy is two phase. The two-phase condition permits the structure to be refined by the α to β to α transformation process on heating and cooling. The process of heating to a high temperature to promote subsequent transformation is known as solution heat treatment. By permitting some beta to be retained temporarily at lower temperature, the alloy elements enable optimum control of the microstructure during subsequent transformation after cooling from the forging or solution heat treatment temperature when the alloys are “aged” (reheated after rapid cooling to temperatures well below the β transus). The α–β alloys, when properly treated, have an excellent combination of strength and ductility. They are stronger than the α or the β-alloys.

The β-alloys are metastable; that is, they tend to transform to an equilibrium, or balance, of structures. The β-alloys generate their strength from the intrinsic strength of the β structure and the precipitation of alpha and other phases from the alloy through heat treatment after processing. The most significant benefit provided by a beta structure is the increased formability of such alloys relative to the hexagonal crystal structure types (α and α–β).

Titanium aluminides differ from conventional titanium alloys in that they are principally chemical compounds alloyed to enhance strength, formability, etc. The aluminides have higher operational temperatures than conventional titanium but at higher cost and, generally, have lower ductility and formability.

In addition to alloys, titanium is sold and used in (CP) forms usually identified as grades. Pure titanium usually has some amount of oxygen alloyed with it. The strength of CP titanium is affected by the interstitial (oxygen and nitrogen) element content. A principal difference among grades is the oxygen (and nitrogen) content, which influences mechanical properties. Small additions of some alloy elements such as palladium are added for increased corrosion resistance in certain grades. A summary of the compositions of many commercial and semicommercial titanium grades and alloys is given in Table 2.

Table 2 Some Commercial and Semicommercial Grades and Alloys of Titanium

	Tensile Strength (min)		0.2% Yield Strength (min)		Impurity Limits wt. % (max)					Nominal Composition, wt. %				
Designation	MPa	ksi	MPa	ksi	N	C	H	Fe	O	Al	Sn	Zr	Mo	Others
Unalloyed grades														
ASTM grade 1	240	35	170	25	0.03	0.08	0.015	0.20	0.18	—	—	—	—	—
ASTM grade 2	340	50	280	40	0.03	0.08	0.015	0.30	0.25	—	—	—	—	—
ASTM grade 3	450	65	380	55	0.05	0.08	0.015	0.30	0.35	—	—	—	—	—
ASTM grade 4	550	80	480	70	0.05	0.08	0.015	0.50	0.40	—	—	—	—	—
ASTM grade 7	340	50	280	40	0.03	0.08	0.015	0.30	0.25	—	—	—	—	0.2Pd
ASTM grade 11	240	35	170	25	0.03	0.08	0.015	0.20	0.18	—	—	—	—	0.2Pd
α and near-α alloys														
Ti–0.3Mo–0.8Ni	480	70	380	55	0.03	0.10	0.015	0.30	0.25	—	—	—	0.3	0.8Ni
Ti–5Al–2.5Sn	790	115	760	110	0.05	0.08	0.02	0.50	0.20	5	2.5	—	—	—
Ti–5Al–2.5Sn–ELI	690	100	620	90	0.07	0.08	0.0125	0.25	0.12	5	2.5	—	—	—
Ti–8Al–1Mo–1V	900	130	830	120	0.05	0.08	0.015	0.30	0.12	8	—	—	1	1V
Ti–6Al–2Sn–4Zr–2Mo	900	130	830	120	0.05	0.05	0.0125	0.25	0.15	6	2	4	2	0.08Si
Ti–6Al–2Nb–1Ta–0.8Mo	790	115	690	100	0.02	0.03	0.0125	0.12	0.10	6	—	—	1	2Nb, 1Ta
Ti–2.25Al–11Sn–5Zr–1Mo	1000	145	900	130	0.04	0.04	0.008	0.12	0.17	2.25	11	5	1	0.2Si
Ti–5.8Al–4Sn–3.5Zr–0.7Nb–0.5Mo–0.35Si	1030	149	910	132	0.03	0.08	0.006	0.05	0.15	5.8	4	3.5	0.5	0.7Nb, 0.35Si
α–β alloys														
Ti–6Al–4V[a]	900	130	830	120	0.05	0.10	0.0125	0.30	0.20	6	—	—	—	4V
Ti–6Al–4V–ELI[a]	830	120	760	110	0.05	0.08	0.0125	0.25	0.13	6	—	—	—	4V
Ti–6Al–6V–2Sn[a]	1030	150	970	140	0.04	0.05	0.015	1.0	0.20	6	2	—	—	0.7Cu, 6V
Ti–8Mn[a]	860	125	760	110	0.05	0.08	0.015	0.50	0.20	—	—	—	—	8.0Mn
Ti–7Al–4Mo[a]	1030	150	970	140	0.05	0.10	0.013	0.30	0.20	7.0	—	—	4.0	—
Ti–6Al–2Sn–4Zr–6Mo[b]	1170	170	1100	160	0.04	0.04	0.0125	0.15	0.15	6	2	4	6	—
Ti–5Al–2Sn–2Zr–4Mo–4Cr[b,c]	1125	163	1055	153	0.04	0.05	0.0125	0.30	0.13	5	2	2	4	4Cr
Ti–6Al–2Sn–2Zr–2Mo–2Cr[c]	1030	150	970	140	0.03	0.05	0.0125	0.25	0.14	5.7	2	2	2	2Cr, 0.25Si
Ti–3Al–2.5V[d]	620	90	520	75	0.015	0.05	0.015	0.30	0.12	3	—	—	—	2.5V
Ti–4Al–4Mo–2Sn–0.5Si	1100	160	960	139	[e]	0.02	0.0125	0.20	[e]	4	2	—	4	0.5Si

Table 2 (*Continued*)

	Tensile Strength (min)		0.2% Yield Strength (min)		Impurity Limits wt. % (max)					Nominal Composition, wt. %				
Designation	MPa	ksi	MPa	ksi	N	C	H	Fe	O	Al	Sn	Zr	Mo	Others
β alloys														
Ti–10V–2Fe–3Al[a,c]	1170	170	1100	160	0.05	0.05	0.015	2.5	0.16	3	—	—	—	10V
Ti–13V–11Cr–3Al[b]	1170	170	1100	160	0.05	0.05	0.025	0.35	0.17	3	—	—	—	11.0Cr, 13.0V
Ti–8Mo–8V–2Fe–3Al[b,c]	1170	170	1100	160	0.03	0.05	0.015	2.5	0.17	3	—	—	8.0	8.0V
Ti–3Al–8V–6Cr–4Mo–4Zr[a,c]	900	130	830	120	0.03	0.05	0.20	0.25	0.12	3	—	4	4	6Cr, 8V
Ti–11.5Mo–6Zr–4.5Sn[a]	690	100	620	90	0.05	0.10	0.020	0.35	0.18	—	4.5	6.0	11.5	—
Ti–15V–3Cr–3Al–3Sn	1000[b] 1241[f]	145[b] 180[f]	965[b] 1172[f]	140[b] 170[f]	0.05	0.05	0.015	0.25	0.13	3	3	—	—	15V, 3Cr
Ti–15Mo–3Al–2.7Nb–0.2Si	862	125	793	115	0.05	0.05	0.015	0.25	0.13	3	—	—	15	2.7Nb, 0.2Si

[a] Mechanical properties given for the annealed condition; may be solution treated and aged to increase strength.

[b] Mechanical properties given for the solution-treated-and-aged condition; alloy not normally applied in annealed conditions.

[c] Semicommercial alloy; mechanical properties and composition limits subject to negotiation with suppliers.

[d] Primarily a tubing alloy; may be cold drawn to increase strength.

[e] Combined $O_2 + 2N_2 = 0.27\%$.

[f] Also solution treated and aged using an alternative aging temperature (480°C, or 900°F)

Source: From *Titanium: A Technical Guide,* 2nd ed., ASM International, Materials Park, OH 44073-0002, 2001, p. 8.

4.2 Strengthening of Titanium Alloys

Desired mechanical properties such as yield or ultimate strength density (strength efficiency), perhaps creep and creep-rupture strength, as well as fatigue-crack growth rate, fracture toughness, and manufacturing considerations such as welding and forming requirements, are extremely important. They normally provide the criteria that determine the alloy composition, structure (α, α–β, or β), heat treatment (some variant of either annealing or solution treating and aging), and level of process control selected or prescribed for structural titanium alloy applications.

By introducing atoms, phases, grain boundaries, or other interfaces into titanium, the movement of imperfections that cause deformation to occur is inhibited. Modification of composition and microstructure enables titanium alloys to be strengthened significantly. Final strength is a function of composition and the various deformation processes used to form and strengthen alloys. It is quite important for the alloy selector to have a realistic understanding of the strengthening process in titanium alloys as the properties of titanium and its alloys can be modified considerably not only by chemistry modification but also by processing.

The titanium alloys derive their strength from a fineness of microstructure produced by transformation of crystal structures in grains from β to α, plus dispersion of one phase in another as in the case of precipitation of α-phases from retained β in metastable beta alloys. The fine structure produced by transformations can often be martensitic in nature, as temperatures on the component being produced are reduced during cooling from deformation processing or solution treatment. The reader may recall that martensitic structures are produced in steels (and in other systems) and can create very strong and hard alloys. Martensitic reactions are found in titanium alloys; they are not as effective as those in steels at causing hardening but do bring about microstructure refinements and thus strength improvements in titanium alloys. Fine dispersions in the alloys usually are produced by "aging" through reheating and holding at an intermediate temperature after prior forging and heat treatment processing.

5 EFFECTS OF ALLOY ELEMENTS

Alloy elements generally can be classified as α stabilizers or β stabilizers. Alpha stabilizers, such as aluminum, oxygen, and nitrogen increase the temperature at which the α-phase is stable. On the other hand, beta stabilizers, such as vanadium and molybdenum, result in stability of the β-phase at lower temperatures. The transformation temperature from α plus β or from α to all β is known as the β transus temperature. The β transus is defined as the lowest equilibrium temperature at which the material is 100% β. The β transus is critical in deformation processing and in heat treatment, as described below. By reference to the β transus, heat treatment temperatures can produce specific microstructures during the heat treatment process. See, for example, the amount of α-phase that can be produced by temperature location relative to the β transus for Ti–6Al–4V as shown in Fig. 4.

5.1 Intermetallic Compounds and Other Secondary Phases

Intermetallic compounds and transient secondary phases are formed in titanium alloy systems along with microstructural variants of the traditional β- and α-

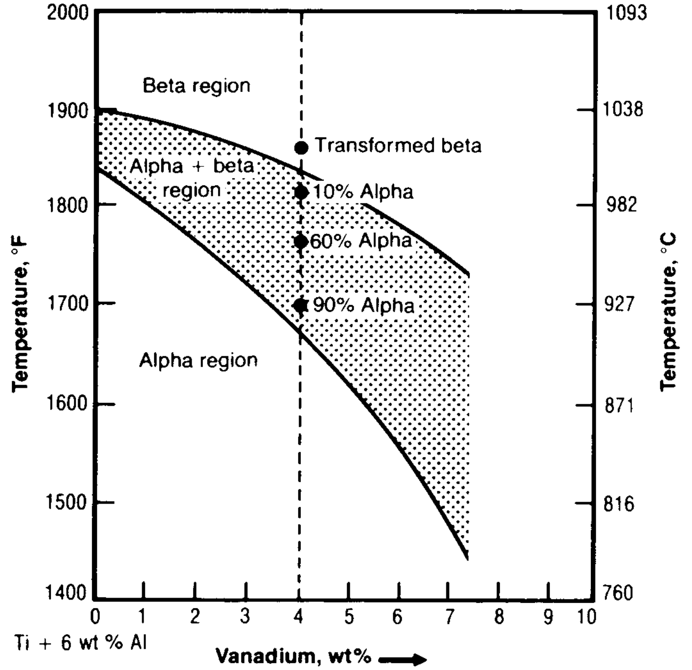


Fig. 4 Phase diagram that predicts the results of heat treatment or forging practice. (From *Titanium: A Technical Guide,* 1st ed., ASM International, Materials Park, OH 44073-0002, 1988, p. 51.)

phases. The more important secondary phases, historically, have been ω and α-2, chemically written as Ti_3Al. Omega phase has not proven to be a factor in commercial systems using present-day processing practice. Alpha-2 has been considered to be a concern in some cases of stress–corrosion cracking. Alloys with extra-high aluminum (favoring α-2) were found to be prone to stress–corrosion cracking. Most present interest in α-2 centers on its use as a matrix for a high-temperature titanium alloy. Another phase more likely to succeed as a high-temperature alloy matrix is γ TiAl, mentioned previously. Gamma-phase is not a factor in the property behavior of conventional titanium alloys.

5.2 Mechanical and Physical Properties

Titanium is a low-density element (approximately 60% of the density of steel and superalloys) that can be strengthened greatly by alloying and deformation processing. The physical and mechanical properties of elemental titanium are given in Table 3. Titanium is nonmagnetic and has good heat-transfer properties. Its coefficient of thermal expansion is somewhat lower than that of steel's and less than half that of aluminum.

Titanium's modulus can vary with alloy type (β vs. α) and processing from as low as about 93 GPa (13.5×10^6 psi) up to about 120.5 GPa (17.5×10^6 psi). For reference, titanium alloy moduli on average are about 50% greater than the moduli for aluminum alloys but only about 50%–60% of the moduli for steels and nickel-base superalloys. Titanium alloys can have their crystals ori-

Table 3 Physical and Mechanical Properties of Elemental Titanium

Atomic number	22
Atomic weight	47.90
Atomic volume	10.6 W/D
Covalent radius	1.32 Å
First ionization energy	158 kcal/g-mol
Thermal neutron absorption cross section	5.6 barns/atom
Crystal structure	• Alpha: close-packed, hexagonal ≤882.5°C (1620°F) • Beta: body-centered, cubic ≥882.5°C (1620°F)
Color	Dark gray
Density	4.51 g/cm^3 (0.163 lb/in.^{3})
Melting point	1668 ± 10°C (3035°F)
Solidus/liquidus	1725°C
Boiling point	3260°C (5900°F)
Specific heat (at 25°C)	0.518 J/kg °K (0.124 Btu/lb · °F)
Thermal conductivity	9.0 Btu/h ft^2 °F
Heat of fusion	440 kJ/kg (estimated)
Heat of vaporization	9.83 MJ/kg
Specific gravity	4.5
Hardness	HRB 70 to 74
Tensile strength	35 ksi min
Modulus of elasticity	14.9×10^6 psi
Young's modulus of elasticity	116×10^9 N/m^2 16.8×10^6 lbf/in.^{2} 102.7 GPa
Poisson's ratio	0.41
Coefficient of friction	0.8 at 40 m/min (125 ft/min) 0.68 at 300 m/min (1000 ft/min)
Specific resistance	554 $\mu\Omega \cdot$ mm
Coefficient of thermal expansion	8.64×10^{-6}/°C
Electrical conductivity	3% IACS (copper 100%)
Electrical resistivity	47.8 $\mu\Omega \cdot$ cm
Electronegativity	1.5 Pauling's
Temperature coefficient of electrical resistance	0.0026/°C
Magnetic susceptibility	1.25×10^{-6} 3.17 emu/g
Machinability rating	40

Source: From *Titanium: A Technical Guide,* 1st ed., ASM International, Materials Park, OH 44073-0002, 1988, p. 11.

ented by processing such that a texture develops. When that happens, instead of the usual random orientation of grains leading to uniformity of mechanical properties, a nonuniform orientation occurs and leads to a greater range of values than expected. By appropriate processing, it should be possible to orient wrought titanium for optimum elastic modulus at the high end of the modulus values quoted above. Although textures can be produced, processing that leads to directional grain or crystal orientation similar to directional solidification in castings or directional recrystallization in oxide dispersion strengthened alloys is not practical in titanium alloy systems.

5.3 Effects of Processing

Properties of titanium alloys generally are controlled by variations of the processing (including heat treatment) and are modified for optimum fatigue resistance by surface treatments such as shot peening. Process treatments can produce either acicular or equiaxed microstructures in most titanium alloys if phase transformations from α to β to α or related phases are permitted to occur. Microstructures have been identified that show α as acicular or equiaxed and with varying amounts of α-phase. The platelike or acicular α produced by transformation from β-phase has special aspects as far as properties are concerned. Table 4 shows the relative behavior of equiaxed versus platelike alpha. No one microstructure is good for all applications.

5.4 Hydrogen (in CP Titanium)

The solubility of hydrogen in α titanium at 300°C (572°F) is about 8 at. % (about 0.15 wt. %, or about 1000 ppm by weight). Hydrogen in solution has little effect on the mechanical properties. Damage is caused by hydrides which form. Upon precipitation of the hydride, the ductility suffers. Hydrogen damage of titanium and titanium alloys, therefore, is manifested as a loss of ductility (embrittlement) and/or a reduction in the stress intensity threshold for crack propagation. Figure 5 shows the effect of hydrogen on reduction of area, a measure of ductility.

No embrittlement is found at 20 ppm hydrogen, which corresponds to about 0.1 at. % of hydrogen. Other data shows that, independent of the heat treatment, this low a concentration has little effect on the impact strength (a different measure of embrittlement). However, as little as 0.5 at. % hydrogen (about 100 atom ppm) can cause measurable embrittlement. Slow cooling from the α region—e.g. 400°C (752°F)—allows sufficient hydride to precipitate to reduce the impact energy. The only practical approach to control the hydrogen problem is to maintain a low concentration of the element. As a result, CP titanium will usually have a maximum allowable hydrogen content of about 0.015 wt. % (about 100 ppm by weight). For example, for the grades of commercially pure

Table 4 Relative Advantages of Equiaxed and Acicular Microstructures

Equiaxed
- Higher ductility and formability
- Higher threshold stress for hot-salt stress corrosion
- Higher strength (for equivalent heat treatment)
- Better hydrogen tolerance
- Better low-cycle fatigue (initiation) properties

Acicular
- Superior creep properties
- Higher fracture-toughness values

Source: From *Titanium: A Technical Guide,* 1st ed., ASM International, Materials Park, OH 44073-0002, 1988, p. 168.

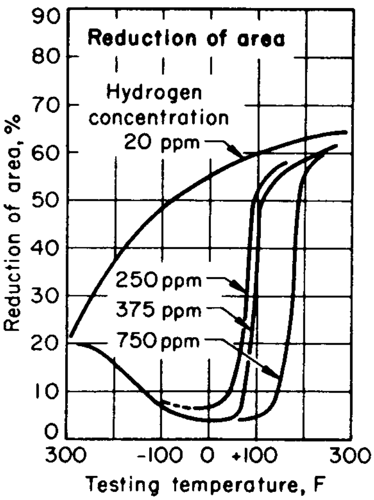


Fig. 5 Ductility of alpha titanium versus test temperature, showing embrittling effects of hydrogen. (From *Titanium: A Technical Guide,* 1st ed., ASM International, Materials Park, OH 44073-0002, 1988, p. 161.)

titanium, the level is about 0.01%. Hydrogen also can have a potent effect on titanium alloy properties.

5.5 Oxygen and Nitrogen (in CP Titanium)

Oxygen and nitrogen have a significant effect on strength properties. As the amount of oxygen and nitrogen increases, the toughness decreases until the material eventually becomes quite brittle. Embrittlement occurs at a concentration considerably below the solubility limit. The allowed oxygen content is higher than the allowed nitrogen content. Yield and ultimate strengths increase as oxygen (and nitrogen) levels go up. The higher strengths in CP titanium grades come from higher oxygen levels. Oxygen (and nitrogen) can have a potent effect on titanium alloy properties as well.

5.6 Mechanical Properties of Titanium Alloys

The grain size, grain shape, and grain boundary arrangements in titanium have a very significant influence on mechanical properties, and it is the ability to manipulate the phases/grains present as a result of alloy composition that is responsible for the variety of properties that can be produced in titanium and its alloys. Transformed β-phase products in alloys can affect tensile strengths, ductility, toughness, and cyclic properties. To these effects must be added the basic strengthening effects of alloy elements.

Interstitial elements are those elements such as oxygen that are significantly smaller than the titanium atom and so may dissolve in the titanium phase crystal lattice as solid solutions without substituting for titanium atoms. Of course, some interstitial elements also may form second phases with titanium. As is the case for comparable-size elements, interstitial elements may have a preference for one phase over another in titanium. As indicated above, a significant influence on mechanical behavior of CP titanium is brought about by hydrogen, nitrogen, carbon, and oxygen, which dissolve interstitially in titanium and have a potent effect on mechanical properties. These effects carry over to titanium alloys in varying degrees.

The ELI (extra-low interstitial) levels specified for some titanium alloys implicitly recognize the effect of reduced interstitials on ductility. ELI-type material is used for critical applications where enhanced ductility and toughness are produced by keeping interstitials at a very low level. Hydrogen is always kept at a low level to avoid embrittlement, yet there still remains concern about the most reasonable level to specify in both CP and alloyed titanium to protect against embrittlement but keep manufacturing cost low.

Although data are not provided here for grain size effects on titanium grades, it is generally accepted that fineness of structure (smaller particle size, grain size, etc.) is more desirable from the point of view of TYS in metallic materials. The UTS is not particularly affected by grain size, but ductility as represented by elongation or reduction in area generally is improved with smaller grain sizes. Ductility is a measure of toughness, but toughness is not normally at issue in CP titanium grades. Another measure of toughness is Charpy impact strength. Chemistry and minimum tensile properties for various specifications for CP and modified titanium grades at room temperature are given in Table 5.

Elevated temperature behavior of titanium grades has been studied, but titanium grades are not customarily used at high temperatures. The near-α or α–β alloys are the preferred materials where high-temperature mechanical properties are desired. With allowance for grain size effects and possible minor chemistry variations, cast CP titanium materials should behave in much the same way as wrought.

Alpha Alloys

Alpha alloys such as Ti–5Al–2.5Sn, Ti–6Al–2Sn–4Zr–2Mo + Si and Ti–8Al–1Mo–1V (see Table 2) are used primarily in gas turbine applications. Ti–8Al–1Mo–1V alloy and Ti–6Al–2Sn–4Zr–2Mo + Si are useful at temperatures above the normal range for the workhorse α–β alloy, Ti–6Al–4V. Ti–8Al–1Mo–1V and Ti–6Al–2Sn–4Zr–2Mo + Si alloys have better creep resistance than Ti–6Al–4V, and creep resistance is enhanced with a fine acicular (Widmanstatten) structure. In its normal heat-treated condition, Ti–6Al–2Sn–4Zr–2Mo + Si alloy actually has a structure better described as α–β.

Alpha and near-α alloys therefore are usually employed in the solution annealed and stabilized condition. Solution annealing may be done at a temperature some 35°C (63°F) below the β transus temperature while stabilization is commonly produced by heating for 8 h at about 590°C (1100°F). These alloys are more susceptible to the formation of ordered Ti_3Al, which promotes stress–corrosion cracking (SCC).

Alpha–Beta Alloys

The most important titanium alloy is the α–β alloy, Ti–6Al–4V. This alloy has found application for a wide variety of aerospace components and fracture-critical parts. With a strength-to-density ratio of 25 × 106 mm (1 × 10^6 in.),

Table 5 CP and Modified Ti: Minimum Room Temperature Tensile Properties for Various Specifications

					Tensile Properties[a]				
	Chemical Composition (% max)				Tensile Strength		Yield Strength		Minimum Elongation (%)
Designation	C	O	N	Fe	MPa	ksi	MPa	ksi	
JIS Class 1	—	0.15	0.05	0.20	275–410	40–60	165[b]	24[b]	27
ASTM grade 1 (UNS R50250)	0.10	0.18	0.03	0.20	240	35	170–310	25–45	24
DIN 3.7025	0.08	0.10	0.05	0.20	295–410	43–60	175	25.5	30
GOST BT1-00	0.05	0.10	0.04	0.20	295	43	—	—	20
BS 19-27t/in.^{2}	—	—	—	0.20	285–410	41–60	195	28	25
JIS Class 2	—	0.20	0.05	0.25	343–510	50–74	215[b]	31[b]	23
ASTM grade 2 (UNS R50400)	0.10	0.25	0.03	0.30	343	50	275–410	40–60	20
DIN 3.7035	0.08	0.20	0.06	0.25	372	54	245	35.5	22
GOST BTI-0	0.07	0.20	0.04	0.30	390–540	57–78	—	—	20
BS 25-35t/in.^{2}	—	—	—	0.20	382–530	55–77	285	41	22
JIS Class 3	—	0.30	0.07	0.30	480–617	70–90	343[b]	50[b]	18
ASTM grade 3 (UNS R50500)	0.10	0.35	0.05	0.30	440	64	377–520	55–75	18
ASTM grade 4 (UNS R50700)	0.10	0.40	0.05	0.50	550	80	480	70	15
DIN 3.7055	0.10	0.25	0.06	0.30	460–590	67–85	323	47	18
ASTM grade 7 (UNS R52400)	0.10	0.25	0.03	0.30	343	50	275–410	40–60	20
ASTM grade 11 (UNS R52250)	0.10	0.18	0.03	0.20	240	35	170–310	25–45	24
ASTM grade 12 (UNS R53400)	0.10	0.25	0.03	0.30	480	70	380	55	12

[a] Unless a range is specified, all listed values are minimums.

[b] Only for sheet, plate, and coil.

Source: From *Materials Property Handbook—Titanium,* ASM International, Materials Park, OH 44073-0002, 1994, p. 224.

Ti–6Al–4V is an effective lightweight structural material and has strength-toughness combinations between those of steel and aluminum alloys. High-strength α–β alloys include Ti–6Al–6V–2Sn and Ti–6Al–2Sn–4Zr–6Mo. Alpha is the dominant phase in all of these alloys but is dominant to a lesser extent in the high-strength alloys than in Ti–6Al–4V. These high-strength alloys are stronger and more readily heat treated than Ti–6Al–4V.

When α–β titanium alloys are heat treated high in the α–β range, and then cooled, the resulting structure, because of the presence of globular (equiaxed) primary α in the transformed β (platelike) matrix is called equiaxed. When a 100% transformed β structure is achieved by cooling from above the β transus, the structure may be called acicular, or needlelike. Generally speaking, α–β alloys would be annealed just below the β transus to produce a maximum of transformed acicular β with approximately 10% of equiaxed α present. Some titanium alloys—for example, Ti–6A1–2Sn–4Zr–2Mo—are given β heat treatments to enhance high–temperature creep resistance. (Castings and powder products may be given a β anneal, too, in order to break up the structure, although not necessarily for optimizing creep strength.)

In actual components, the structure of titanium α–β type of alloys is controlled not only by how much working is done and by how close to, or above, the β transus the alloy is processed, but also by the section size of the component. Ideally, alloys should have good hardenability, i.e., ability to reach desired cooling rates and attendant microstructures in fairly thick sections. Many α–β alloys do not have great hardenability. Ti–6Al–4V alloy only has sufficient hardenability to be effectively heat treated to full property levels in sections less than 25 mm (1 in.) thick.

One of the least understood concepts in the behavior of α–β titanium alloys is that of aging. With few exceptions titanium alloys do not age in the classical sense—that is, where a secondary, strong intermetallic compound appears and strengthens the matrix by its dispersion. A dispersion is produced, on aging of α–β alloys, but it is thought to be β dispersed in the α or martensitic alpha prime. Beta is not materially different from α phase with respect to strength; however, the effectiveness of strengthening in titanium alloys appears to center in the number and fineness of α–β phase boundaries. Annealing and rapid cooling, which maximize α–β boundaries for a fixed primary α content, along with aging, which may promote additional boundary structure, can significantly increase alloy strength.

Beta Alloys

An alloy is a β-alloy if it contains sufficient β stabilizer alloying element to retain the β-phase without transformation to martensite on quenching to room temperature. A number of titanium alloys (see Table 2) contain more than this minimum amount of β stabilizer alloy addition. The more highly β stabilized alloys are alloys such as Ti–3Al–8V–6Cr–4Mo–4Zr (beta C) and Ti–15V–3Cr–3Al–3Sn. Solute-lean β alloys are sometimes classified as β-rich α–β alloys, and this class includes Ti–10V–2Fe–3Al and proprietary alloys such as Ti–17 (Ti–5Al–2Sn–2Zr–4Mo–4Cr) and beta CEZ (Ti–5Al–2Sn–4Zr–4Mo–2Cr).

In a strict sense there is no truly stable β-alloy because even the most highly alloyed β will, on holding at elevated temperatures, begin to precipitate ω, α,

Ti_3Al, or silicides, depending on temperature, time, and alloy composition. All β-alloys contain a small amount of aluminum, an α stabilizer, to strengthen α that may be present after heat treatment. The composition of the precipitating α is not constant and will depend on the temperature of heat treatment. The higher the temperature in the α–β phase field, the higher will be the aluminum content of α.

The processing window is tighter than that normally used for the other alloy types (α and α–β alloys). For the less highly β stabilized alloys, such as Ti–10V–2Fe–3Al, for example, the thermomechanical process is critical to the property combinations achieved as this has a strong influence on the final microstructure and the resultant tensile strength and fracture toughness that may be achieved. Exacting control of thermomechanical processing is somewhat less important in the more highly β stabilized alloys, such as Ti–3Al–8V–6Cr–4Mo–4Zr and Ti–15V–3Cr–3Al–3Sn. In these alloys, the final microstructure, precipitated α in the β phase, is so fine that microstructural manipulation through thermomechanical processing is not as effective.

Properties

Wrought Alloys. Typical room temperature property values for titanium alloy mill products are listed in Table 6. Fractions of room temperature strength retained at elevated temperatures by the same titanium alloys are shown in Table 7. Data for unalloyed titanium are included in Table 7 to illustrate that the alloys not only have higher room temperature strengths than unalloyed titanium but also retain much larger fractions of that strength at elevated temperatures. Typical tensile strengths and 0.1% creep strengths as functions of temperature of some selected alloys are shown in Figs. 6 and 7, respectively.

Fatigue life in unalloyed titanium depends on grain size, interstitial (oxygen, etc.) level, and degree of cold work, as illustrated in Fig. 8. A decrease in grain size in unalloyed titanium from 110 μm down to 6 μm improves the 10^7 cycle fatigue endurance limit by 30%. HCF endurance limits of unalloyed titanium depend on interstitial contents just as do the YS and UTS. The ratio of HCF endurance limit and YS at ambient temperature appears to remain relatively constant as YS changes with interstitial content but does show a temperature dependence.

There are significant differences among titanium alloys in fracture toughness, but there is also appreciable overlap in their properties. Table 8 gives examples of typical plane–strain fracture toughness ranges for α–β titanium alloys. From these data it is apparent that the basic alloy chemistry affects the relationship between strength and toughness. From Table 8 it is also evident, as noted earlier, that transformed microstructures may greatly enhance toughness while only slightly reducing strength. It is well known that toughness depends on thermomechanical processing (TMP) to provide the desired structure. However, the enhancement of fracture toughness at one stage of an operation—for example, a forging billet—does not necessarily carry over to a forged part. Because welds in alloy Ti–6Al–4V contain transformed products, one would expect such welds to be relatively high in toughness. This is, in fact, the case. In addition to welding, many other factors such as environment, cooling rates in large sections (i.e.,

Table 6 Tensile Strengths of Several Commercial Titanium-Base Alloys: Typical Room Temperature Values

Alloy Name	Nominal Composition	Condition	Tensile Strength		Yield Strength		Elongation (%)
			ksi	10^8 N/m^2	ksi	10^8 N/m^2	
5-2.5	Ti–5Al–2.5Sn	Annealed (0.25–4 h/1300–1600°F)	120–130	8.3–9.0	115–120	7.9–8.3	13–18
3-2.5	Ti–3Al–2.5V	Annealed (1–3 h/1200–1400°F)	95	6.5	90	6.2	22
6-2-1-1	Ti–6Al–2Nb–1Ta–1Mo	Annealed (0.25–2 h/1300–1700°F)	125	8.6	110	7.6	14
8-1-1	Ti–8Al–1Mo–1V	Annealed (8 h/1450°F)	145	10.0	135	9.3	12
Corona 5	Ti–4.5Al–5Mo–1.5Cr	α–β annealed after β processing	140–160	9.7–11.0	135–150	9.3–10.3	12–15
Ti-17	Ti–5Al–2Sn–2Zr–4Mo–4Cr	α–β or β processed plus aged	165	11.4	155	10.7	8
6-4	Ti–6Al–4V	Annealed (2 h/1300–1600°F)	140	9.6	130	9.0	17
		Aged	170	11.7	160	11.0	12
6-6-2	Ti–6Al–6V–2Sn	Annealed (3 h/1300–1500°F)	155	10.7	145	10.0	14
		Aged	185	12.8	175	12.1	10
6-2-4-2	Ti–6Al–2Sn–4Zr–2Mo	Annealed (4 h/1300–1550°F)	145	10.0	135	9.3	15
6-2-4-6	Ti–6Al–2Sn–4Zr–6Mo	Annealed (2 h/1500–1600°F)	150	10.3	140	9.7	11
		Aged	175	12.1	165	11.4	8
6-22-22	Ti–6Al–2Sn–2Zr–2Mo–2Cr–0.25Si	α–β processed plus aged	162	11.2	147	10.1	14
10-2-3	Ti–10V–2Fe–3Al	Annealed (1 h/1400°F)	140	9.7	130	9.0	9
		Aged	180–195	12.4–13.4	165–180	11.4–12.4	7
15-3-3-3	Ti–15V–3Cr–3Sn–3Al	Annealed (0.25 h/1450°F)	115	7.9	112	7.7	20–25
		Aged	165	11.4	155	10.7	8
13-11-3	Ti–13V–11Cr–3Al	Annealed (0.5 h/1400–1500°F)	135–140	9.3–9.7	125	8.6	18
		Aged	175	12.1	165	11.4	7
38-6-44	Ti–3Al–8V–6Cr–4Mo–4Zr	Annealed (0.5 h/1500–1700°F)	120–130	8.3–9.0	113–120	7.8–8.3	10–15
		Aged	180	12.4	170	11.7	7
β-III	Ti–4.5Sn–6Zr–11.5Mo	Annealed (0.5 h/1300–1600°F)	100–110	6.9–7.6	95	6.5	23
		Aged	180	12.4	170	11.7	7

Source: From *Materials Property Handbook—Titanium,* ASM International, Materials Park, OH 44073-0002, 1994, p. 106.

Table 7 Fraction of Room Temperature Strength Retained at Elevated Temperature for Several Titanium Alloys[a]

Temperature		Unalloyed Ti		Ti–6Al–4V		Ti–6Al–6V–2Sn		Ti–6Al–2Sn–4Zr–6Mo		Ti–6Al–2Sn–4Zr–2Mo		Ti-1100[a]		IMI-834	
°C	°F	TS	YS	TS	YS	TS	YS	TS	YS	TS	YS	TS	YS	TS	YS
93	200	0.80	0.75	0.90	0.87	0.91	0.89	0.90	0.89	0.93	0.90	0.93	0.92	—	—
204	400	0.57	0.45	0.78	0.70	0.81	0.74	0.80	0.80	0.83	0.76	0.81	0.85	0.85	0.78
316	600	0.45	0.31	0.71	0.62	0.76	0.69	0.74	0.75	0.77	0.70	0.76	0.79	—	—
427	800	0.36	0.25	0.66	0.58	0.70	0.63	0.69	0.71	0.72	0.65	0.75	0.76	—	—
482	900	0.33	0.22	0.60	0.53	—	—	0.66	0.69	0.69	0.62	0.72	0.74	—	—
538	1000	0.30	0.20	0.51	0.44	—	—	0.61	0.66	0.66	0.60	0.69	0.69	—	—
593	1100	—	—	—	—	—	—	—	—	—	—	0.66	0.63	0.63	0.61

[a] Short time tensile test with less than 1 h at temperature prior to test.

TS = tensile strength; YS = yield strength.

Source: From *Fatigue Data Handbook: Light Structural Alloys,* ASM International, Materials Park, OH 44073-0002, p. 189.

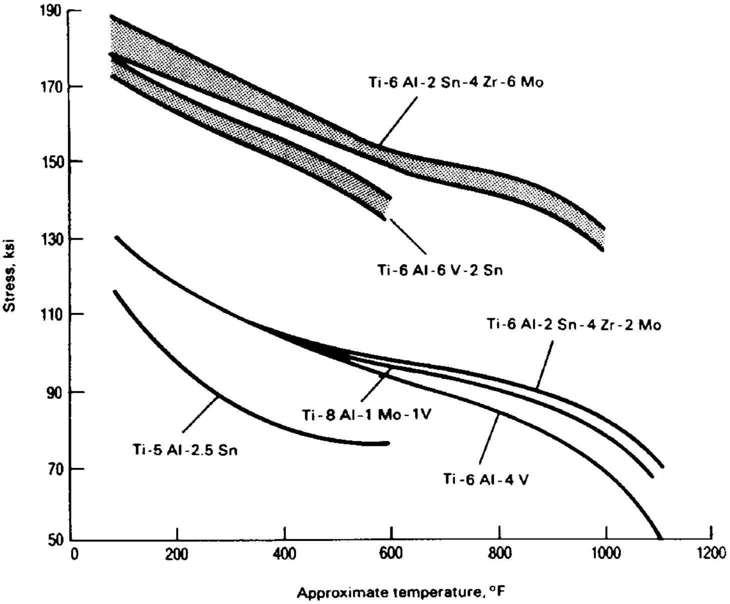


Fig. 6 Comparison of typical ultimate tensile strengths of selected titanium alloys as a function of temperature. (From *Titanium: A Technical Guide,* 1st ed., ASM International, Materials Park, OH 44073-0002, 1988, p. 173.)

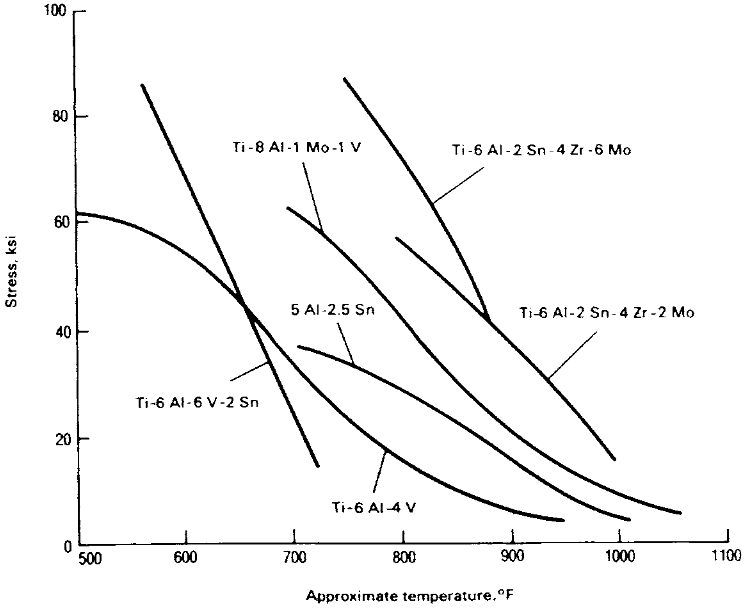


Fig. 7 Comparison of typical 150-h, 0.1% creep strengths for selected titanium alloys. (From *Titanium: A Technical Guide,* 1st ed., ASM International, Materials Park, OH 44073-0002, 1988, p. 174.)

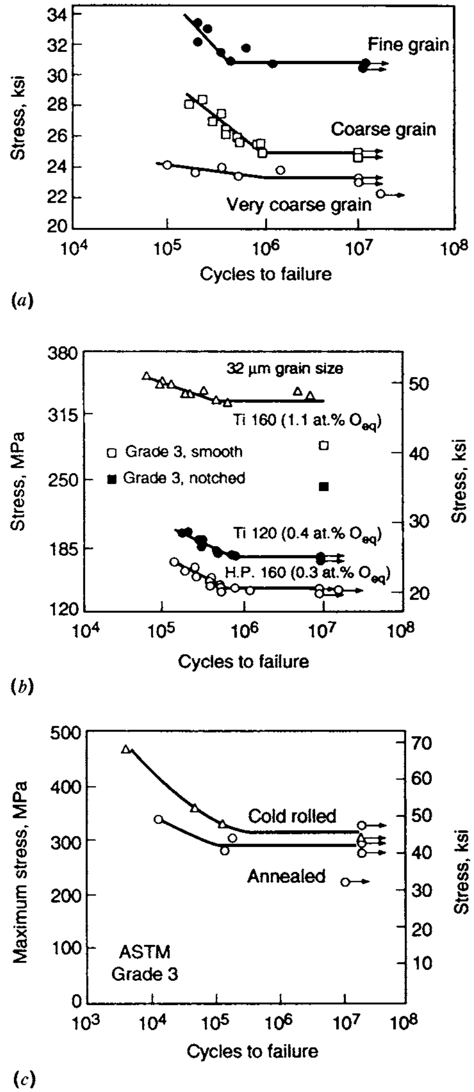


Fig. 8 Stress versus cycles-to-failure curves for pure titanium as affected by (*a*) grain size (*b*) oxygen content, and (*c*) cold work. (From *Metals Handbook, Vol. 19,* ASM International, Materials Park, OH 44073-0002, 1996, p. 837.)

hardenability, a factor that affects structural fineness), hydrogen content, etc. may affect K_{Ic}.

Titanium alloys may show less resistance to notches than other alloys. Notch strength in fatigue is significantly lower than smooth strength. Scratches on the surfaces of titanium alloy components can lead to reduced fatigue capability. High levels of favorable compressive residual stresses usually exist in titanium alloys as a result of machining. These levels are sometimes enhanced by surface processing such as glass bead or shot peening.

Table 8 Typical Fracture Toughness Values of High-Strength Titanium Alloys

Alloy	Alpha Morphology	Yield Strength		Fracture Toughness K_{Ic}	
		MPa	ksi	$MPa \cdot m^{1/2}$	$ksi \cdot in.^{1/2}$
Ti–6Al–4V	Equiaxed	910	130	44–66	40–60
	Transformed	875	125	88–110	80–100
Ti–6Al–6V–2Sn	Equiaxed	1085	155	33–55	30–50
	Transformed	980	140	55–77	50–70
Ti–6Al–2Sn–4Zr–6Mo	Equiaxed	1155	165	22–23	20–30
	Transformed	1120	160	33–55	30–50

Source: From *Titanium: A Technical Guide,* 1st ed., ASM International, Materials Park, OH 44073-0002, 1988, p. 168.

Cast Alloys. Cast-titanium alloys are generally α–β alloys. They are equal, or nearly equal, in strength to wrought alloys of the same compositions. Typical room temperature tensile properties of several cast-titanium alloys are shown in Table 9 while creep strength of cast Ti–6Al–4V is shown in Table 10. Virtually all existing data have been generated from alloy Ti–6Al–4V; consequently, the basis for most cast-alloy property data is Ti–6Al–4V. Because the microstructure of cast-titanium alloy parts is comparable to that of wrought material, many properties of cast plus HIP parts are at similar levels to those for wrought alloys. These properties include tensile strength, creep strength, fracture toughness, and fatigue crack propagation.

Generally, castings of titanium alloys are hot isostatically pressed (HIP) to close casting porosity. HIP conditions may affect the resultant properties as HIP is just another heat treatment as far as microstructure is concerned. It also should be noted that test results are often on small separately cast test coupons and will

Table 9 Typical Room Temperature Tensile Properties of Several Cast-Titanium Alloys (bars machined from castings)[a]

Alloy[b,c]	Yield Strength		Tensile Strength		Elongation (%)	Reduction of area (%)
	MPa	ksi	MPa	ksi		
Commercially pure (grade 2)	448	65	552	80	18	32
Ti–6Al–4V, annealed	855	124	930	135	12	20
Ti–6Al–4V–ELI	758	110	827	120	13	22
Ti–1100, beta-STA[d]	848	123	938	136	11	20
Ti–6Al–2Sn–4Zr–2Mo, annealed	910	132	1006	146	10	21
IMI-834, beta-STA[d]	952	138	1069	155	5	8
Ti–6Al–2Sn–4Zr–6Mo, beta-STA[d]	1269	184	1345	195	1	1
Ti–3Al–8V–6Cr–4Zr–4Mo, beta-STA[d]	1241	180	1330	193	7	12
Ti–15V–3Al–3Cr–3Sn, beta-STA[d]	1200	174	1275	185	6	12

[a] Specification minimums are less than these typical properties.

[b] Solution-treated and aged (STA) heat treatments can be varied to produce alternate properties.

[c] ELI, extra low interstitial.

[d] Beta-STA, soluton treatment within β-phase field followed by aging.

Source: Metals Handbook, Vol. 2, ASM International, Materials Park, OH 44073-0002, p. 637.

Table 10 Ti–6Al–4V: Creep Strength of Cast Material

Test Temperature		Stress		Plastic Strain on Loading	Test Duration	Time, h, to Reach Creep of		
°C	°F	MPa	ksi	(%)	(h)	0.1%	0.2%	1.0%
455	850	276	40.0	0	611.2	2.0	9.6	610.0
425	800	276	40.0	0	500.0	15.0	60.0	—
425	800	345	60.0	0	297.5	3.5	11.0	291.5
400	750	448	65.0	0.7	251.4	7.5	22.0	—
370	700	414	60.0	0.3	500	240.0	—	—
315	600	517	75.0	2.04	330.9	0.02	0.04	0.1
260	500	534	77.5	2.1	307.9	0.01	0.02	0.1
205	400	552	80.0	0.56	138.0	0.1	0.13	1.5
205	400	531	77.0	0.8	18.2	0.02	0.04	0.16
175	350	517	75.0	0.01	1006.0	0.4	2.2	—
150	300	517	75.0	—	500	0.25	1.2	—
150	300	517	75.0	—	500	1.7	12.2	—
120	250	517	75.0	0.0	1006.1	9.8	160.0	—

Note: Specimens from hubs of centrifugal compressor impellers that were cast, HIPed (2 h at 900°C, or 1650°F), and 103.5 MPa, 15.0 ksi, and aged 1.5 h at 675°C (1250°F). Specimen blanks approximately 5.72 by 0.95 by 0.96 cm (2.25 by 0.37 by 0.37 in.) in section size, with the long axis oriented tangential to the hub section, were machined to standard-type creep specimens 3.81 mm (0.150 in.) in diameter. The specimens were lathe turned and then polished with 320-grit emery paper. The creep-rupture tests were performed at 120–455°C (250–850°F) using dead-load-type creep frames in air over a stress ranged of 276–552 MPa (40–80 ksi). The microstructure consisted of transformed β grains with discontinuous grain-boundary α and colonies of transformed β that contained packets of parallel-oriented α platelets separated by a thin layer of aged β.

Source: A. Chakrabarti and E. Nichols, Creep Behavior of Cast Ti–6Al–4V Alloy, *Titanium '80: Science and Technology,* Proceedings of the 4th International Conference on Titanium, Kyoto, Japan, 19–22 May 1980, Vol. 2, H. Kimura and O. Izumi, Ed., TMS/AIME, 1980, pp. 1081–1096.

not necessarily reflect the property level achievable with similar processing on a full-scale cast part. Property levels of actual cast parts, especially larger components, probably will be somewhat lower, the result of coarser grain structure or slower quench rates achieved.

Summary. Powder metallurgy technology has been applied to titanium alloy processing with limited success, partially owing to economic issues. Wrought processing remains the preferred method of achieving shape and property control. Cast alloy processing is used but for a limited alloy base. Figure 9 shows fatigue scatter bands for wrought, cast, and powder metallurgy products of Ti–6Al–4V alloy for comparison of attainable properties.

6 MANUFACTURING PROCESSES

6.1 General Aspects of the Manufacture of Titanium Articles

Appropriate compositions of titanium can be forged, rolled to sheet, or otherwise formed into a variety of shapes. Some compositions can be processed as large investment castings. Commercial large castings are made mostly in the titanium alloy Ti–6Al–4V, which has been in production for over 40 years. Fabricated titanium structures can be built up by welding or brazing. Fabricated structures are primarily made with Ti–6Al–4V. Fabricated structures may contain cast as

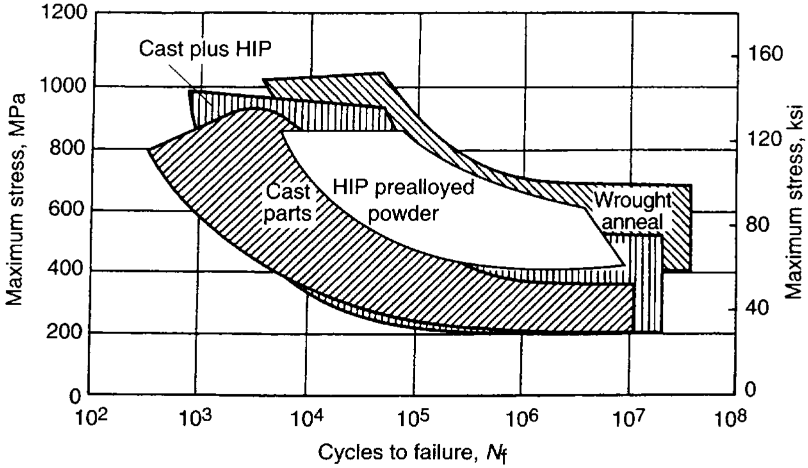


Fig. 9 Fatigue scatter bands for ingot metallurgy, castings, and powder metallurgy products of Ti–6Al–4V alloy. (From *Titanium: A Technical Guide,* 2nd ed., ASM International, Materials Park, OH 44073-0002, 2001, p. 116.)

well as wrought parts, although wrought components are used for most applications.

Single-piece forged gas turbine fan and compressor disks are prime applications for titanium alloys. Titanium wrought, cast, and powder metallurgy products find use in the biomedical arena. Fan blades and compressor blades of titanium represent areas that continue to receive support despite the reported threat from composites. By and large, most titanium alloys are wrought, in particular, forged.

The manufacture of titanium alloys consists of a number of separate steps of which the following represent the transfer of titanium from an ore to an ingot ready for either wrought or cast processing or to mill products:

- Production of titanium sponge (reduction of titanium ore to an impure porous form of titanium metal)
- Purification of the sponge
- Melting of sponge or sponge plus alloy elements or a master alloy to form an electrode
- Remelting and, possibly, remelting again to homogenize the first electrode and create an ingot for further processing
- Primary fabrication, in which ingots are converted into billets or general mill products such as bar, plate, sheet, strip, or wire
- Secondary fabrication where a billet or bar may be forged into an approximate final shape

6.2 Production of Titanium via Vacuum Arc Melting

Whether the final product is to be a forged or investment cast one, the essence of a titanium alloy's ability to create the properties desired hinges on the correct application of melting principles. Melting practices may be classified as either primary (the initial melt of elemental materials and/or scrap which sets the composition) or secondary (remelt, often more than once, of a primary melt for the purpose of controlling the solidification structure). The melt type or combination of melt types selected depends upon the alloy composition, mill form and size desired, properties desired, and sensitivity of the final component to localized inhomogeneity in the alloy.

The principal method for the production of titanium electrodes and ingots since commercial introduction of titanium alloys occurred in the 1950s has been the use of vacuum arc remelting (VAR). The purity of the titanium alloys produced is a function of the purity of the starting materials. Control of raw materials is extremely important in producing titanium and its alloys because there are many elements of which even small amounts can produce major, and at times undesirable, effects on the properties of these metals in finished form. To produce ingots of titanium or its alloys for commercial application, titanium from sponge is commonly alloyed with pure other elements, master melt of titanium plus alloy elements, and/or reclaimed titanium scrap (usually called "revert").

Because sponge is an uneven product consisting of a loose, granular mass, it does not compact as well as might be desired in some instances. Compacting is needed to make an electrode from which to melt the alloy. During melting, a piece of the sponge might fall unmelted into the solidifying electrode. Perhaps a chunk of revert or master melt might fall in. Whatever the situation, a gross inhomogeneity would result. Depending on the type and size of the inhomogeneity, a major structural defect could exist. Consequently, after some significant incidents in aircraft gas turbine engines about 30–40 years ago, second and then third melts were instituted to provide almost certain homogenization of the alloy.

Defects have been a concern for titanium ingot metallurgy production since the early days of the industry. Different types of defects were recognized, most stemming from sponge handling, electrode preparation, and melt practice. The principal characterization of these defects was as low-density inclusions (LDI) and high-density inclusions (HDI). Over two dozen different defects have been cataloged. Defects prompted strict process controls that were agreed upon jointly by metal suppliers and customers alike. These controls have done much to attain either reduced-defect or defect-free materials. Despite the controls, occasional defects have been involved in significant events of titanium-alloy-related failures. It is predicted that the introduction of cold-hearth technologies will further reduce the incidence of defects in titanium ingots. Electron beam and plasma arc melting technologies are now available for the melting of titanium alloys or the remelting of scrap. The use of these technologies permits the controlled hearth melting (CHM) of titanium alloys. Studies on electron-beam cold-hearth melting (EBCHM) and plasma arc melting (PAM) demonstrated the ability of hearth melting to remove HDI with great confidence. LDI are also addressed by CHM.

6.3 Forging Titanium Alloys

Manufacturing processes such as die forging, hot and cold forming, machining, chemical milling, joining, and sometimes, extrusion are all secondary fabrication processes used for producing finished parts from billet or mill products. Each of these processes may strongly influence properties of titanium and its alloys, either alone or by interacting with effects of processes to which the metal has previously been subjected. Titanium alloy forgings are produced by all the forging methods currently available. These include open-die, closed-die, rotary forging, and others. One of the main purposes of forging, in addition to shape control, is to obtain a combination of mechanical properties that generally does not exist in bar or billet. Tensile strength, creep resistance, fatigue strength, and toughness all may be better in forgings than in bar or other forms. Selection of the optimal forging method is based on the shape desired for the forged component as well as its desired mechanical properties and microstructure (which largely determines properties after alloy composition is set).

Open-die forging is used to produce some shapes in titanium when volume and/or size do not warrant the development of closed dies for a particular application. However, closed-die forging is used to produce the greatest amount of forged titanium alloys. Closed-die forging can be classified as blocker-type (single die set), conventional (two or more die sets), or high definition (two or more die sets). Precision die forging is also conducted, usually employing hot-die/isothermal forging techniques. Conventional closed-die titanium forgings cost more than the blocker-type, but the increase in cost is justified because of reduced machining costs and better property control. The dies used in titanium forging are heated to facilitate the forging process and to reduce surface chilling and metal temperature losses that can lead to inadequate die filling and/or excessive surface cracking of the forged component. Hot-die/isothermal forging takes the die temperature to higher levels.

Forging is more than just a shape-making process. The key to successful forging and heat treatment is the β transus temperature. Fundamentally, there are two principal approaches to the forging of titanium alloys:

- Forging the alloy predominantly below the β transus
- Forging the alloy predominantly above the β transus

Conventional α–β forging is best described as a forging process in which all or most of the deformation is conducted below the β transus. Alpha, β and transformed β-phases will be present in the microstructure at some time. Structures resulting from α–β forging are characterized by deformed or equiaxed primary α-phase (α present during the forging process) and transformed β phase (acicular in appearance). Beta forging is a forging technique in which the deformation of the alloy is done above the β transus. In commercial practice, β forging actually involves initial deformation above the β transus but final finishing with controlled amounts of deformation below the β transus of the alloy. In β forging, the influences of mechanical working (deformation) are not necessarily cumulative because of the high temperature and because of the formation of new grains by recrystallization each time the β transus is surpassed on reheating for forging. Beta forging, particularly of α and α–β alloys, results in significant reductions in forging pressures and reduced cracking tendency of the billet during forging.

An alternative titanium die forging procedure involves the use of precision isothermal (sometimes superplastic) forging techniques. Precision isothermal forging produces a product form that requires much less machining than conventionally forged alloy to achieve final dimensions of the component. Precision-forged titanium is a significant factor in titanium usage in the aircraft and gas turbine engine field. Most precision-forged titanium is produced as near-net shape (NNS) products, meaning that the forging is close to final dimensions but that some machining is required.

Superplastic forming, a variant of superplastic isothermal forging, currently is widely used in the aircraft industry and to a lesser extent is used in the gas turbine industry. Advantages of superplastic forming are, among others:

- Very complex parts can be formed.
- Lighter, more efficient structures can be designed and formed.
- It is performed in a single operation.
- More than one piece may be produced in a machine cycle.
- Pressure (force) is uniformly applied to all areas of the workpiece.

Superplastic forming coupled with diffusion bonding (SPFDB) has been used on titanium alloys to produce complex fabricated structures.

6.4 Investment Casting

Cost factors associated with wrought alloy processing led to continual efforts to develop and improve casting methods for titanium and its alloys. The result has been a somewhat checkered application of titanium castings with a more widespread acceptance of the practice in the last decade of the twentieth century. Titanium castings now are used extensively in the aerospace industry and to lesser measure in the chemical process, marine, biomedical, automotive, and other industries. The investment casting process uses a disposable mold to create a negative image of the desired component. Metal fills the mold and solidifies with the desired shape and dimensions very close to final desired values. Some machining is necessary.

Several alloys were studied at first, but soon investigators concentrated on Ti–6Al–4V with results that supported an idea that cast-titanium parts could be made with strength levels and characteristics approaching those of conventional wrought alloys. Subsequently, additional titanium components have successfully been cast from pure titanium, α–β and β-alloys. Nonetheless, the primary alloy used for casting of titanium components is Ti–6Al–4V. Some important concepts to remember are:

- Hot isostatic pressing may be required to close casting porosity.
- Heat treatment to develop properties may require close monitoring.
- Cast component properties will tend to fall in the lower end of the scatterband for wrought versions of the alloy chosen (Ti–6Al–4V, unlikely that any other conventional alloy will be cast).
- Section thickness may affect properties generated in castings.

6.5 Machining and Residual Stresses

Machining of titanium alloys is similar to but more difficult than that of machining stainless steels. In welding or machining of titanium alloys, the effects of the energy input (heat energy, deformation energy) on the microstructure and properties of the final product must be considered, just as it must be done in forging. Favorable residual stresses have been generated on titanium surfaces for years. Properties measured will degrade dramatically if the favorable surface residual stresses are reduced, for example, by chemical polishing. Shot peening is a favorite method of increasing a titanium alloy's fatigue strength, at least in airfoil roots and other nongas path regions.

6.6 Joining

Components of titanium alloys are routinely welded. Titanium and most titanium alloys can be joined by the following fusion welding techniques:

- Gas–tungsten arc welding (GTAW)
- Gas–metal arc welding (GMAW)
- Plasma arc welding (PAW)
- Electron beam welding (EBW)
- Laser beam welding (LBW)

and by brazing or such solid-state joining techniques as diffusion bonding, inertia bonding, and friction welding.

Just as occurs in the heat treatment of titanium and its alloys, fusion welding processes can lead to pickup of detrimental gases. Alloys must be welded in such a way as to preclude interstitial gases such as oxygen from being incorporated in the weld or weld-heat-affected area. For successful arc welding of titanium and titanium alloys, complete shielding of the weld is necessary because of the high reactivity of titanium to oxygen and nitrogen at welding temperatures. Excellent welds can be obtained in titanium and its alloys in a welding chamber, where welding is done in a protective gas atmosphere, thus giving adequate shielding. When welding titanium and titanium alloys, only argon or helium, and occasionally a mixture of these two gases, are used for shielding. Since it is more readily available and less costly, argon is more widely used. Welding in a chamber, however, is not always practical. Open-air techniques can be used with fusion welding when the area to be joined is well shielded by an inert gas using a Mylar bag for gas containment. Such atmospheric control by means of a temporary bag, or chamber, is preferred. Because titanium alloy welds are commonly used in fatigue-critical applications, a stress relief operation is generally required following welding to minimize potentially detrimental residual stresses. The essence of joining titanium and its alloys is adhering to the following principle conditions that need to be met:

- Detrimental interstitial elements must be excluded from the joint region.
- Contaminants (scale, oil, etc.) must be excluded from the joint region.
- Detrimental phase changes must be avoided to maintain joint ductility.

When proper techniques are developed and followed, the welding of thin-to-moderate section thickness in titanium alloys can be accomplished successfully using all of the aforementioned processes. For welding titanium thicker than about 2.54 mm (0.10 in.) by the GTAW process, a filler metal must be used.

For PAW, a filler metal may or may not be used for welding metal less than 12.7 mm (0.5 in.) thick.

Titanium and its alloys can be brazed. Argon, helium, and vacuum atmospheres are satisfactory for brazing titanium. For torch brazing, special fluxes must be used on the titanium. Fluxes for titanium are primarily mixtures of fluorides and chlorides of the alkali metals, sodium, potassium, and lithium. Vacuum and inert-gas atmospheres protect titanium during furnace and induction-brazing operations. Titanium assemblies frequently are brazed in high-vacuum, cold–wall furnaces. A vacuum of 10^{-3} torr, or more, is required to braze titanium. Ideally, brazing should be done in a vacuum at a pressure of about 10^{-5}–10^{-4} torr or done in a dry inert-gas atmosphere if vacuum brazing is not possible.

7 OTHER ASPECTS OF TITANIUM ALLOY SELECTION

7.1 Corrosion

Although titanium and its alloys are used chiefly for their desirable mechanical properties, among which the most notable is their high strength-to-weight ratio, another important characteristic of the metal and its alloys is titanium's outstanding resistance to corrosion. CP titanium offers excellent corrosion resistance in most environments, except those media that contain fluoride ions. Unalloyed titanium is highly resistant to the corrosion normally associated with many natural environments, including seawater, body fluids, and fruit and vegetable juices. Titanium exposed continuously to seawater for about 18 years has undergone only superficial discoloration. Titanium is more corrosion resistant than stainless steel in many industrial environments, and its use in the chemical process industry has been continually increasing. Titanium exhibits excellent resistance to atmospheric corrosion in both marine and industrial environments.

The major corrosion problems with titanium alloys appear to be crevice corrosion, which occurs in locations where the corroding media are virtually stagnant. Pits, if formed, may progress in a similar manner. Other problem areas are with a potential for stress–corrosion, particularly at high temperatures, resulting in hot-salt stress–corrosion cracking (HSSCC). HSSCC has been observed in experimental testing and an occasional service failure. Stress–corrosion cracking (SCC) is a fracture, or cracking, phenomenon caused by the combined action of tensile stress, a susceptible alloy, and a specific corrosive environment. Another important characteristic of SCC is the requirement that tensile stress be present. Aluminum additions increase susceptibility to SCC; alloys containing more than 6% Al generally are susceptible to stress–corrosion.

HSSCC of titanium alloys is a function of temperature, stress, and time of exposure In general, HSSCC has not been encountered at temperatures below about 260°C (500°F). The greatest susceptibility occurs at about 290–425°C (about 550–800°F) based on laboratory tests. Time-to-failure decreases as either temperature or stress level is increased. All commercial alloys, but not unalloyed titanium, have some degree of susceptibility to HSSCC. Alpha alloys are more susceptible than other alloys.

7.2 Biomedical Applications

Titanium alloys have become standards in the orthopedic industry where hip implants, for example, benefit from several characteristics of titanium:

- Excellent resistance to corrosion by body fluids
- High specific strength owing to good mechanical strengths and low density
- Modulus about 50–60% of that of competing cobalt-base superalloys

Corrosion resistance benefits would seem to be evident. High specific strength, however, enables a lighter implant to be made with attendant improvement in patient response to the device. Lastly, the modulus of bone is very low, about 10% of that of stainless steel or cobalt-base alloys. The degree of load transfer from the implant to the bony structure in which it is implanted and which it replaces is a direct function of the modulus. By reducing the elastic modulus, the bone can be made to receive a greater share of the load. The result with lower modulus titanium alloys is a longer operating time before breakdown of the implant–bone assembly.

7.3 Cryogenic Applications

Many of the available α and α–β titanium alloys have been evaluated at subzero temperatures, but service experience at such temperatures has been gained only for a few alloys. Ti–5Al–2.5Sn and Ti–6Al–4V have very high strength–to–weight ratios at cryogenic temperatures and have been the preferred alloys for special applications at temperatures from −196 to −269°C (−320 to −452°F). Impurities such as iron and the interstitials oxygen, carbon, nitrogen, and hydrogen tend to reduce the toughness of these alloys at both room and subzero temperatures. For maximum toughness, extra-low-interstitial (ELI) grades are specified for critical applications.

8 FINAL COMMENTS

Many titanium alloys have been developed, although the total is small compared to other metals such as steels and superalloys. A principle reason for this situation is the high cost of alloy development and of proving the worth and safety of a new material. In the sport world, titanium made a brief fling at commercial nongas turbine applications when the golf club market virtually tied up titanium metal for a short time in the 1990s. Titanium bicycle frames are marketed but are quite expensive.

The titanium market has been a roller coaster over the years, and gas turbine applications remain the most significant part of the application market. Within most aircraft gas turbine engine companies, only a few alloys have ever made it to production. Admittedly this list differs from company to company in the United States and somewhat more with alloys used outside the United States. Nevertheless, it is apparent that, although the ability to push titanium's operating environment higher in temperature has resulted in significant gains, advances have tapered off. Since the mid-1950s when Pratt & Whitney put the first titanium in U.S. gas turbines, much industrial and government funding has been used to increase alloy capabilities. Titanium aluminides have been the subject of multidecades of study with interesting but hardly commercially viable results. Barring discovery of some unforeseen nature, the message is that, if an existing alloy works and a new alloy does not offer some benefit that overrides the development cost of proving up the alloy for its new use, do not change alloys. For the ever-shrinking cadre of developers in industry, the current status suggests

Table 11 Associations Providing Titanium Information

Titanium Information Group
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FAX: +44 (0) 1562 824851
WEB: www.titaniuminfogroup.co.uk
E-MAIL: rayportman@talk21.com

Japan Titanium Society
22-9 Kanda Nishiki-Cho
Chiyoda-Ku, Tokyo ZIP 101
Japan
TEL: 081 (3) 3293 5958
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WEB: www.titan-japan.com

International Titanium Association
Brian Simpson, Executive Director
350 Interlocken Blvd. Suite 390
Broomfield, CO 80021-3485
TEL: 303 404 2221
FAX: 303 404 9111
WEB: www.titanium.org
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that efforts to tailor existing alloys and "sell" them for new or existing applications may have better return on investment.

If an alloy selector is starting from scratch to pick an alloy for an application, then any commercially available alloy may be fair game. On the other hand, the best alloy may not be available owing to corporate patent protection or insufficient market to warrant its continued production by the limited number of manufacturers. Then, selection of another alloy from a producer may be necessary but may possibly require development costs to get the product in workable form and to determine design properties. If possible, select a known alloy that has more than one supplier and more than one casting or forging source. In all likelihood, unless a special need (such as formability of sheet) or maximum high-temperature strength is required, Ti–6Al–4V might be the first choice. For special needs such as in marine applications or biomedical orthopedic situations, choice of other alloys may be warranted. In any event, one should work with the suppliers and others in the manufacturing chain to acquire typical or design properties for the alloy in the form it will be used. Generic alloys owned by melters or developers are best for the alloy selector not associated with one of the big corporate users of titanium alloys. Companies with proprietary interests usually have nothing to benefit from giving up a technological advantage by sharing design data or even granting a production release to use a proprietary alloy. Table 11 lists a few organizations chartered to provide assistance to users of titanium products. A list of suppliers should be available from them.

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CHAPTER 7

NICKEL AND ITS ALLOYS

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1 INTRODUCTION

Nickel, the 24th element in abundance, has an average content of 0.016% in the outer 10 miles of the earth's crust. This is greater than the total for copper, zinc, and lead. However, few of these deposits scattered throughout the world are of commercial importance. Oxide ores commonly called laterites are largely distributed in the tropics. The igneous rocks contain high magnesium contents and have been concentrated by weathering. Of the total known ore deposits, more than 80% is contained in laterite ores. The sulfide ores found in the northern hemispheres do not easily concentrate by weathering. The sulfide ores in the Sudbury district of Ontario, which contain important by-products such as copper, cobalt, iron, and precious metals are the world's greatest single source of nickel.[1]

Nickel has an atomic number of 28 and is one of the transition elements in the fourth series in the periodic table. The atomic weight is 58.71 and density is 8.902 g/cm^3. Useful properties of the element are the modulus of elasticity and its magnetic and magnetostrictive properties, and high thermal and electrical conductivity. Hydrogen is readily adsorbed on the surface of nickel. Nickel will also adsorb other gases such as carbon monoxide, carbon dioxide, and ethylene. It is this capability of surface adsorption of certain gases without forming stable compounds that makes nickel an important catalyst.[2]

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Handbook of Materials Selection. Edited by Myer Kutz

As an alloying element, nickel is used in hardenable steels, stainless steels, special corrosion-resistant and high-temperature alloys, copper–nickel, "nickel–silvers," and aluminum–nickel. Nickel imparts ductility and toughness to cast iron.

Approximately 10% of the total annual production of nickel is consumed by electroplating processes. Nickel can be electrodeposited to develop mechanical properties of the same order as wrought nickel; however, special plating baths are available that will yield nickel deposits possessing a hardness as high as 450 Vickers (425 BHN). The most extensive use of nickel plate is for corrosion protection of iron and steel parts and zinc-base die castings used in the automotive field. For these applications, a layer of nickel, 0.0015–0.003 in. thick, is used. This nickel plate is then finished or covered with a chromium plate consisting in thickness of about 1% of the underlying nickel plate thickness in order to maintain a brilliant, tarnish-free, hard exterior surface.

2 NICKEL ALLOYS

Most of the alloys listed and discussed are in commercial production. However, producers from time to time introduce improved modifications that make previous alloys obsolete. For this reason, or economic reasons, they may remove certain alloys from their commercial product line. Some of these alloys have been included to show how a particular composition compares with the strength or corrosion resistance of currently produced commercial alloys.

2.1 Classification of Alloys

Nickel and its alloys can be classified into the following groups on the basis of chemical composition.[3]

Nickel

(1) Pure nickel, electrolytic (99.56% Ni), carbonyl nickel powder and pellet (99.95% Ni); (2) commercially pure wrought nickel (99.6–99.97% nickel); and (3) anodes (99.3% Ni).

Nickel and Copper

(1) Low-nickel alloys (2–13% Ni); (2) cupronickels (10–30% Ni); (3) coinage alloy (25% Ni); (4) electrical resistance alloy (45% Ni); (5) nonmagnetic alloys (up to 60% Ni); and (6) high-nickel alloys, Monel (over 50% Ni).

Nickel and Iron

Wrought alloy steels (0.5–9% Ni); (2) cast alloy steels (0.5–9% Ni); (3) alloy cast irons (1–6 and 14–36% Ni); (4) magnetic alloys (20–90% Ni): (a) controlled coefficient of expansion (COE) alloys (29.5–32.5% Ni) and (b) high-permeability alloys (49–80% Ni); (5) nonmagnetic alloys (10–20% Ni); (6) clad steels (5–40% Ni); (7) thermal expansion alloys: (a) low expansion (36–50% Ni) and (b) selected expansion (22–50% Ni).

Iron, Nickel, and Chromium

(1) Heat-resisting alloys (40–85% Ni); (2) electrical resistance alloys (35–60% Ni); (3) iron-base superalloys (9–26% Ni); (4) stainless steels (2–25% Ni); (5) valve steels (2–13% Ni); (6) iron-base superalloys (0.2–9% Ni); (7) maraging steels (18% Ni).

Nickel, Chromium, Molybdenum, and Iron

(1) Nickel-base solution-strengthened alloys (40–70% Ni); (2) nickel-base precipitation-strengthened alloys (40–80% Ni).

Powder-Metallurgy Alloys

(1) Nickel-base dispersion strengthened (78–98% Ni); (2) nickel-base mechanically alloyed oxide-dispersion-strengthened (ODS) alloys (69–80% Ni).

The nominal chemical composition of nickel-base alloys is given in Table 1. This table does not include alloys with less than 30% Ni, cast alloys, or welding products. For these and those alloys not listed, the chemical composition and applicable specifications can be found in the *Unified Numbering System for Metals and Alloys*, published by the Society of Automotive Engineers, Inc.

2.2 Discussion and Applications

The same grouping of alloys used in Tables 1, 2, and 3, which give chemical composition and mechanical properties, will be used for discussion of the various attributes and uses of the alloys as a group. Many of the alloy designations are registered trademarks of producer companies.

Nickel Alloys

The corrosion resistance of nickel makes it particularly useful for maintaining product purity in the handling of foods, synthetic fibers, and caustic alkalies, and also in structural applications where resistance to corrosion is a prime consideration. It is a general-purpose material used when the special properties of the other nickel alloys are not required. Other useful features of the alloy are its magnetic and magnetostrictive properties; high thermal and electrical conductivity; low gas content; and low vapor pressure.[4]

Typical *nickel 200* applications are food-processing equipment, chemical shipping drums, electrical and electronic parts, aerospace and missile components, caustic handling equipment and piping, and transducers.

Nickel 201 is preferred to nickel 200 for applications involving exposure to temperatures above 316°C (600°F). Nickel 201 is used as coinage, plater bars, and combustion boats in addition to some of the applications for Nickel 200.

Permanickel alloy 300 by virtue of the magnesium content is age-hardenable. But, because of its low alloy content, alloy 300 retains many of the characteristics of nickel. Typical applications are grid lateral winding wires, magnetostriction devices, thermostat contact arms, solid-state capacitors, grid side rods, diaphragms, springs, clips, and fuel cells.

Duranickel alloy 301 is another age-hardenable high nickel alloy, but is made heat treatable by aluminum and titanium additions. The important features of alloy 301 are high strength and hardness, good corrosion resistance, and good spring properties up to 316°C (600°F); and it is on these mechanical considerations that selection of the alloy is usually based. Typical applications are extrusion press parts, molds used in the glass industry, clips, diaphragms, and springs.

Nickel–Copper Alloys

Nickel–copper alloys are characterized by high strength, weldability, excellent corrosion resistance, and toughness over a wide temperature range. They have

Table 1 Nominal Chemical Composition (wt%)

Material	Ni	Cu	Fe	Cr	Mo	Al	Ti	Nb	Mn	Si	C	Other Elements
Nickel												
Nickel 200	99.6	—	—	—	—	—	—	—	0.23	0.03	0.07	—
Nickel 201	99.7	—	—	—	—	—	—	—	0.23	0.03	0.01	—
Permanickel alloy 300	98.7	—	0.02	—	—	—	0.49	—	0.11	0.04	0.29	0.38 Mg
Duranickel alloy 301	94.3	—	0.08	—	—	4.44	0.44	—	0.25	0.50	0.16	—
Nickel–Copper												
Monel alloy 400	65.4	32	1.00	—	—	—	—	—	1.0	0.10	0.12	—
Monel alloy 404	54.6	45.3	0.03	—	—	—	—	—	0.01	0.04	0.07	—
Monel alloy R-405	65.3	31.6	1.25	—	—	0.1	—	—	1.0	0.17	0.15	0.04 S
Monel alloy K-500	65.0	30	0.64	—	—	2.94	0.48	—	0.70	0.12	0.17	—
Nickel–Chromium–Iron												
Inconel alloy 600	76	0.25	8.0	15.5	—	—	—	—	0.5	0.25	0.08	—
Inconel alloy 601	60.5	0.50	14.1	23.0	—	1.35	—	—	0.5	0.25	0.05	—
Inconel alloy 690	60	—	9.0	30	—	—	—	—	—	—	0.01	—
Inconel alloy 706	41.5	0.15	40	16	—	0.20	1.8	3	0.18	0.18	0.03	—
Inconel alloy 718	53.5	0.15	18.5	19	3.0	0.5	0.9	5.1	0.18	—	0.04	—
Inconel alloy X-750	73	0.25	7	15.5	—	0.70	2.5	1	0.50	0.25	0.04	—
Nickel–Iron–Chromium												
Incoloy alloy 800	31	0.38	46	20	—	0.38	0.38	—	0.75	0.50	0.05	—
Incoloy alloy 800H	31	0.38	46	20	—	0.38	0.38	—	0.75	0.50	0.07	—
Incoloy alloy 825	42	1.75	30	22.5	3	0.10	0.90	—	0.50	0.25	0.01	—
Incoloy alloy 925	43.2	1.8	28	21	3	0.35	2.10	—	0.60	0.22	0.03	—
Pyromet 860	44	—	Bal	13	6	1.0	3.0	—	0.25	0.10	0.05	4.0 Co
Refractaloy 26	38	—	Bal	18	3.2	0.2	2.6	—	0.8	1.0	0.03	20 Co
Nickel–Iron												
Nilo alloy 36	36	—	61.5	—	—	—	—	—	0.5	0.09	0.03	
Nilo alloy 42	41.6	—	57.4	—	—	—	—	—	0.5	0.06	0.03	—
Ni–Span–C alloy 902	42.3	0.05	48.5	5.33	—	0.55	2.6	—	0.40	0.50	0.03	—
Incoloy alloy 903	38	—	41.5	—	—	0.90	1.40	2.9	0.09	0.17	0.02	14 Co
Incoloy alloy 907	37.6	0.10	41.9	—	—	1.5	—	4.70	0.05	0.08	0.02	14 Co

Nickel–Chromium–Molybdenum												
Hastelloy alloy X	Bal[c]	—	19	22	9	—	—	—	—	—	0.10	—
Hastelloy alloy G	Bal	2	19.5	22	6.5	—	—	2.1	<1	1.5	<0.05	<1 W, <2.5 Co
Hastelloy alloy C-276	Bal	—	5.5	15.5	16	—	—	—	<0.08	<1	<0.01	2.5 Co, 4 W, 0.35 V
Hastelloy alloy C	Bal	—	<3	16	15.5	—	<0.7	—	<0.08	<1	<0.01	<2 Co
Inconel alloy 617	54	—	—	22	9	1	—	—	—	—	0.07	12.5 Co
Inconel alloy 625	Bal	—	2.5	21.5	9	<0.4	<0.4	3.6	—	—	0.03	—
MAR-M-252	Bal	—	—	19	10	1	2.6	—	<0.5	<0.5	0.15	10 Co, 0.005 B
Rene′ 41	Bal	—	—	19	10	1.5	3.1	—	—	—	0.09	11 Co <0.010 B
Rene′ 95	Bal	—	—	14	3.5	3.5	2.5	3.5	—	—	0.15	8 Co, 3.5 W, 0.01 B, 0.05 Zr
Astroloy	Bal	—	—	15	5.3	4.4	3.5	—	—	—	0.06	15 Co
Udimet 500	Bal	—	<0.5	19	4	3.0	3.0	—	—	—	0.08	18 Co 0.007 B
Udimet 520	Bal	—	—	19	6	2.0	3.0	—	—	—	0.05	12 Co. 1 W, 0.005 B
Udimet 600	Bal	—	<4	17	4	4.2	2.9	—	—	—	0.04	16 Co. 0.02 B
Udimet 700	Bal	—	—	15	5.0	4.4	3.5	—	—	—	0.07	18.5 Co, 0.025 B
Udimet 1753	Bal	—	9.5	16.3	1.6	1.9	3.2	—	0.1	0.05	0.24	7.2 Co, 8.4 W, 0.008 B, 0.06 Zr
Waspaloy	Bal	<0.1	<2	19	4.3	1.5	3	—	—	—	0.08	14 Co, 0.006 B, 0.05 Zr
Nickel-Powder Alloys (*Dispersion Strengthened*)												
TD–nickel	98	—	—	—	—	—	—	—	—	—	—	2 ThO_2
TD–NiCr	Bal	—	—	20	—	—	—	—	—	—	—	1.7 ThO_2
Nickel-Powder Alloys (*Mechanically Alloyed*)												
Inconel alloy MA 754	78	—	1.0	20	—	0.3	0.5	—	—	—	0.05	0.6 Y_2O_3
Inconel alloy MA 6000	69	—	—	15	2	4.5	2.5	—	—	—	0.05	4 W, 2 Ta, 1.1 Y_2O_3

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[a] Minimum.

[b] Maximum.

[c] Balance.

Table 2 Mechanical Properties of Nickel Alloys

Material	0.2% Yield Strength (ksi)[a]	Tensile Strength (ksi)[a]	Elongation (%)	Rockwell Hardness
Nickel				
Nickel 200	21.5	67	47	55 Rb
Nickel 201	15	58.5	50	45 Rb
Permanickel alloy 300	38	95	30	79 Rb
Duranickel alloy 301	132	185	28	36 Rc
Nickel–Copper				
Monel alloy 400	31	79	52	73 Rb
Monel alloy 404	31	69	40	68 Rb
Monel alloy R-405	56	91	35	86 Rb
Monel alloy K-500	111	160	24	25 Rc
Nickel–Chromium–Iron				
Inconel alloy 600	50	112	41	90 Rb
Inconel alloy 601	35	102	49	81 Rb
Inconel alloy 690	53	106	41	97 Rb
Inconel alloy 706	158	193	21	40 Rc
Inconel alloy 718	168	205	20	46 Rc
Inconel alloy X-750	102	174	25	33 Rc
Nickel–Iron–Chromium				
Incoloy alloy 800	48	88	43	84 Rb
Incoloy alloy 800H	29	81	52	72 Rb
Incoloy alloy 825	44	97	53	84 Rb
Incoloy alloy 925	119	176	24	34 Rc
Pyromet 860	115	180	21	37 Rc
Refractaloy 26	100	170	18	—
Nickel–Iron				
Nilo alloy 42	37	72	43	80 Rb
Ni–Span–C alloy 902	137	150	12	33 Rc
Incoloy alloy 903	174	198	14	39 Rc
Incoloy alloy 907	163	195	15	42 Rc
Nickel–Chromium–Molybdenum				
Hastelloy alloy X	52	114	43	—
Hastelloy alloy G	56	103	48.3	86 Rb
Hastelloy alloy C-276	51	109	65	—
Inconel alloy 617	43	107	70	81 Rb
Inconel alloy 625	63	140	51	96 Rb
MAR-M-252	122	180	16	—
Rene′ 41	120	160	18	—
Rene′ 95	190	235	15	—
Astroloy	152	205	16	—
Udimet 500	122	190	32	—
Udimet 520	125	190	21	—
Udimet 600	132	190	13	—
Udimet 700	140	204	17	—
Udimet 1753	130	194	20	39 Rc
Waspaloy	115	185	25	—
Nickel-Powder Alloys (Dispersion Strengthened)				
TD–Nickel	45	65	15	—
TD–NiCr	89	137	20	—
Nickel-Powder Alloys (Mechanically Alloyed)				
Inconel alloy MA 754	85	140	21	—
Inconel alloy MA 6000	187	189	3.5	—

[a] MPa = ksi × 6.895.

Table 3 1000-hr Rupture Stress (ksi)[a]

	1200°F	1500°F	1800°F	2000°F
Nickel–Chromium–Iron				
Inconel alloy 600	14.5	3.7	1.5	—
Inconel alloy 601	28	6.2	2.2	1.0
Inconel alloy 690	16	—	—	—
Inconel alloy 706	85	—	—	—
Inconel alloy 718	85	—	—	—
Inconel alloy X-750	68	17	—	—
Nickel–Iron–Chromium				
Incoloy alloy 800	20	—	—	—
Incoloy alloy 800H	23	6.8	1.9	0.9
Incoloy alloy 825	26	6.0	1.3	—
Pyromet 860	81	17	—	—
Refractaloy 26	65	15.5	—	—
Nickel–Chromium–Moloybdenum				
Hastelloy alloy X	31	9.5	—	—
Inconel alloy 617	52	14	3.8	1.5
Inconel alloy 625	60	7.5	—	—
MAR-M-252	79	22.5	—	—
Rene′ 41	102	29	—	—
Rene′ 95	125	—	—	—
Astroloy	112	42	8	—
Udimet 500	110	30	—	—
Udimet 520	85	33	—	—
Udimet 600	—	37	—	—
Udimet 700	102	43	7.5	—
Udimet 1753	98	34	6.5	—
Waspaloy	89	26	—	—
Nickel-Powder Alloys (Dispersion Strengthened)				
TD–Nickel	21	15	10	7
TD–NiCr	—	—	8	5
Nickel-Powder Alloys (Mechanically Alloyed)				
Inconel alloy MA 754	38	—	19	14
Inconel alloy MA 6000	—	—	22	15

[a] MPa ksi × 6.895.

excellent service in seawater or brackish water under high-velocity conditions, as in propellers, propeller shafts, pump shafts, and impellers and condenser tubes, where resistance to the effects of cavitation and erosion are important. Corrosion rates in strongly agitated and aerated seawater usually do not exceed 1 mil/year.

Monel alloy 400 has low corrosion rates in chlorinated solvents, glass-etching agents, sulfuric and many other acids, and practically all alkalies, and it is resistant to stress-corrosion cracking. Alloy 400 is useful up to 538°C (1000°F) in oxidizing atmospheres, and even higher temperatures may be used if the environment is reducing. Springs of this material are used in corrosive environments up to 232°C (450°F). Typical applications are valves and pumps; pump and propeller shafts; marine fixtures and fasteners; electrical and electronic components; chemical processing equipment; gasoline and freshwater tanks; crude petroleum stills, process vessels, and piping; boiler feedwater heaters and other heat exchangers; and deaerating heaters.

Monel alloy 404 is characterized by low magnetic permeability and excellent brazing characteristics. Residual elements are controlled at low levels to provide a clean, wettable surface even after prolonged firing in wet hydrogen. Alloy 404 has a low Curie temperature and its magnetic properties are not appreciably affected by processing or fabrication. This magnetic stability makes alloy 404 particularly suitable for electronic applications. Much of the strength of alloy 404 is retained at outgassing temperatures. Thermal expansion of alloy 404 is sufficiently close to that of many other alloys as to permit the firing of composite metal tubes with negligible distortion. Typical applications are waveguides, metal-to-ceramic seals, transistor capsules, and power tubes.

Monel alloy R-405 is a free-machining material intended almost exclusively for use as stock for automatic screw machines. It is similar to alloy 400 except that a controlled amount of sulfur is added for improved machining characteristics. The corrosion resistance of alloy R-405 is essentially the same as that of alloy 400, but the range of mechanical properties differs slightly. Typical applications are water meter parts, screw machine products, fasteners for nuclear applications, and valve seat inserts.

Monel alloy K-500 is an age-hardenable alloy that combines the excellent corrosion resistance characteristics of the Monel nickel–copper alloys with the added advantage of increased strength and hardness. Age hardening increases its strength and hardness. Still better properties are achieved when the alloy is cold-worked prior to the aging treatment. Alloy K-500 has good mechanical properties over a wide temperature range. Strength is maintained up to about 649°C (1200°F), and the alloy is strong, tough, and ductile at temperatures as low as −253°C (−423°F). It also has low permeability and is nonmagnetic to −134°C (−210°F). Alloy K-500 has low corrosion rates in a wide variety of environments. Typical applications are pump shafts and impellers, doctor blades and scrapers, oil-well drill collars and instruments, electronic components, and springs.

Nickel–Chromium–Iron Alloys

This family of alloys was developed for high-temperature oxidizing environments. These alloys typically contain 50–80% nickel, which permits the addition of other alloying elements to improve strength and corrosion resistance while maintaining toughness.

Inconel alloy 600 is a standard engineering material for use in severely corrosive environments at elevated temperatures. It is resistant to oxidation at temperatures up to 1177°C (2150°F). In addition to corrosion and oxidation resistance, alloy 600 presents a desirable combination of high strength and workability, and is hardened and strengthened by cold-working. This alloy maintains strength, ductility, and toughness at cryogenic as well as elevated temperatures. Because of its resistance to chloride-ion stress-corrosion cracking and corrosion by high-purity water, it is used in nuclear reactors. For this service, the alloy is produced to exacting specifications and is designated Inconel alloy 600T. Typical applications are furnace muffles, electronic components, heat-exchanger tubing, chemical- and food-processing equipment, carburizing baskets, fixtures and rotors, reactor control rods, nuclear reactor components, primary heat-exchanger tubing, springs, and primary water piping. Alloy 600, being one of the early high-temperature, corrosion-resistant alloys, can be thought of as being the basis of many of our present day special-purpose high-nickel alloys, as illustrated in Fig. 1.

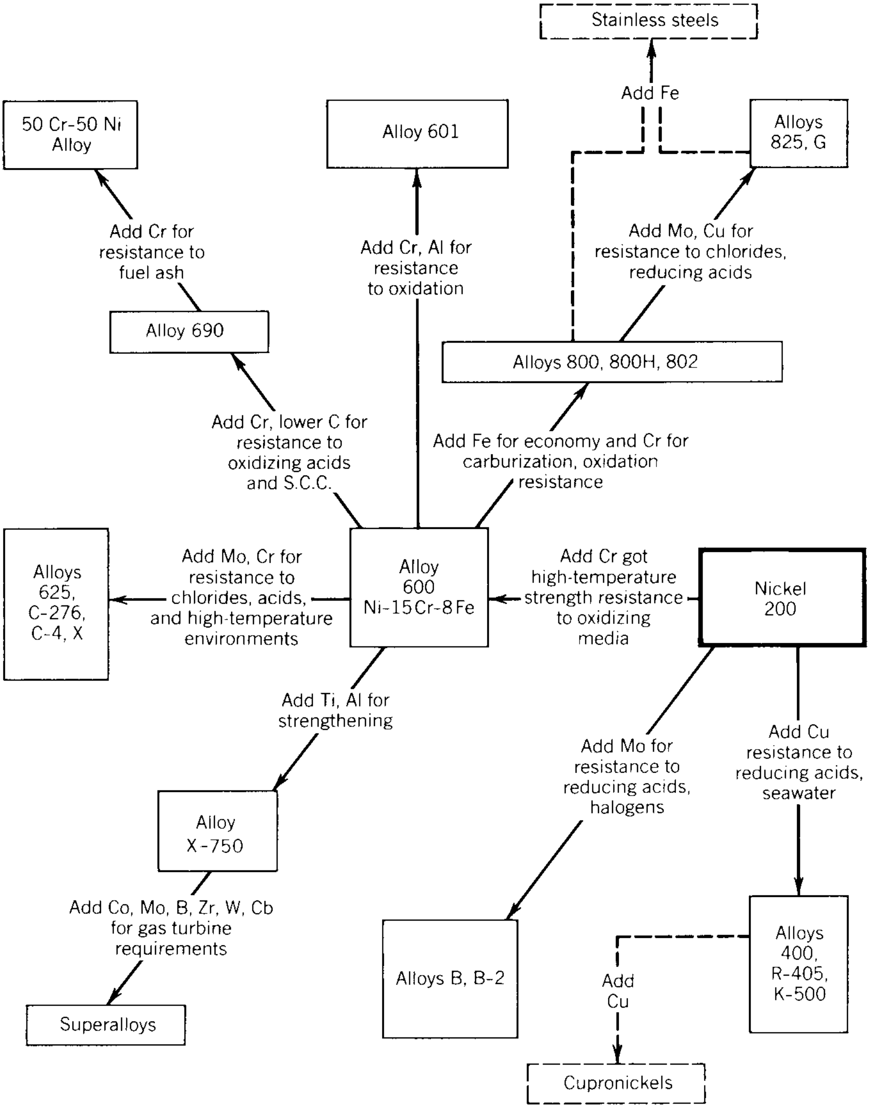

Fig. 1 Some compositional modifications of nickel and its alloys to produce special properties.

Inconel alloy 601 has shown very low rates of oxidation and scaling at temperatures as high as 1093°C (2000°F). The high chromium content (nominally 23%) gives alloy 601 resistance to oxidizing, carburizing, and sulfur-containing environments. Oxidation resistance is further enhanced by the aluminum content. Typical applications are heat-treating baskets and fixtures, radiant furnace tubes, strand-annealing tubes, thermocouple protection tubes, and furnace muffles and retorts.

Inconel alloy 690 is a high-chromium nickel alloy having very low corrosion rates in many corrosive aqueous media and high-temperature atmospheres. In various types of high-temperature water, alloy 690 also displays low corrosion rates and excellent resistance to stress-corrosion cracking—desirable attributes for nuclear steam-generator tubing. In addition, the alloy's resistance to sulfur-containing gases makes it a useful material for such applications as coal-gasification units, burners and ducts for processing sulfuric acid, furnaces for petrochemical processing, and recuperators and incinerators.

Inconel alloy 706 is a precipitation-hardenable alloy with characteristics similar to alloy 718, except that alloy 706 has considerably improved machinability. It also has good resistance to oxidation and corrosion over a broad range of temperatures and environments. Like alloy 718, alloy 706 has excellent resistance to postweld strain-age cracking. Typical applications are gas-turbine components and other parts that must have high strength combined with good machinability and weldability.

Inconel alloy 718 is an age-hardenable high-strength alloy suitable for service at temperatures from −253°C (−423°F) to 704°C (1300°F). The fatigue strength of alloy 718 is high, and the alloy exhibits high stress-rupture strength up to 704°C (1300°F) as well as oxidation resistance up to 982°C (1800°F). It also offers good corrosion resistance to a wide variety of environments. The outstanding characteristic of alloy 718 is its slow response to age hardening. The slow response enables the material to be welded and annealed with no spontaneous hardening unless it is cooled slowly. Alloy 718 can also be repair-welded in the fully aged condition. Typical applications are jet engine components, pump bodies and parts, rocket motors and thrust reversers, and spacecraft.

Inconel alloy X-750 is an age-hardenable nickel–chromium–iron alloy used for its corrosion and oxidation resistance and high creep-rupture strength up to 816°C (1500°F). The alloy is made age-hardenable by the addition of aluminum, columbium, and titanium, which combine with nickel, during proper heat treatment, to form the intermetallic compound $Ni_3(Al, Ti)$. Alloy X-750, originally developed for gas turbines and jet engines, has been adopted for a wide variety of other uses because of its favorable combination of properties. Excellent relaxation resistance makes alloy X-750 suitable for springs operating at temperatures up to about 649°C (1200°F). The material also exhibits good strength and ductility at temperatures as low as −253°C (−423°F). Alloy X-750 also exhibits high resistance to chloride-ion stress-corrosion cracking even in the fully age-hardened condition. Typical applications are gas-turbine parts (aviation and industrial), springs (steam service), nuclear reactors, bolts, vacuum envelopes, heat-treating fixtures, extrusion dies, aircraft sheet, bellows, and forming tools.

Nickel–Iron–Chromium Alloys

This series of alloys typically contains 30−45% Ni and is used in elevated- or high-temperature environments where resistance to oxidation or corrosion is required.

Incoloy alloy 800 is a widely used material of construction for equipment that must resist corrosion, have high strength, or resist oxidation and carburization. The chromium in the alloy imparts resistance to high-temperature oxidation and general corrosion. Nickel maintains an austenitic structure so that the alloy remains ductile after elevated-temperature exposure. The nickel content also contributes resistance to scaling, general corrosion, and stress-corrosion cracking. Typical applications are heat-treating equipment and heat exchangers in the chemical, petrochemical, and nuclear industries, especially where resistance to stress-corrosion cracking is required. Considerable quantities are used for sheathing on electric heating elements.

Incoloy alloy 800H is a version of Incoloy alloy 800 having significantly higher creep and rupture strength. The two alloys have the same chemical composition with the exception that the carbon content of alloy 800H is restricted to the upper portion of the standard range for alloy 800. In addition to a controlled carbon content, alloy 800H receives an annealing treatment that produces a coarse grain size—an ASTM number of 5 or coarser. The annealing treatment and carbon content are responsible for the alloy's greater creep and rupture strength.

Alloy 800H is useful for many applications involving long-term exposure to elevated temperatures or corrosive atmospheres. In chemical and petrochemical processing, the alloy is used in steam/hydrocarbon reforming for catalyst tubing, convection tubing, pigtails, outlet manifolds, quenching-system piping, and transfer piping; in ethylene production for both convection and cracking tubes; in oxo-alcohol production for tubing in hydrogenation heaters; in hydrodealkylation units for heater tubing; and in production of vinyl chloride monomer for cracking tubes, return bends, and inlet and outlet flanges.

Industrial heating is another area of wide usage for alloy 800H. In various types of heat-treating furnaces, the alloy is used for radiant tubes, muffles, retorts, and assorted furnace fixtures. Alloy 800H is also used in power generation for steam superheater tubing and high-temperature heat exchangers in gas-cooled nuclear reactors.

Incoloy alloy 825 was developed for use in aggressively corrosive environments. The nickel content of the alloy is sufficient to make it resistant to chloride-ion stress-corrosion cracking, and, with molybdenum and copper, alloy 825 has resistance to reducing acids. Chromium confers resistance to oxidizing chemicals. The alloy also resists pitting and intergranular attack when heated in the critical sensitization temperature range. Alloy 825 offers exceptional resistance to corrosion by sulfuric acid solutions, phosphoric acid solutions, and seawater. Typical applications are phosphoric acid evaporators, pickling-tank heaters, pickling hooks and equipment, chemical-process equipment, spent nuclear fuel element recovery, propeller shafts, tank trucks, and oil-country cold-worked tubulars.

Incoloy alloy 925 was developed for severe conditions found in corrosive wells containing H_2S, CO_2, and brine at high pressures. Alloy 925 is a weldable, age-hardenable alloy having corrosion and stress-corrosion resistance similar to Incoloy alloy 825. It is recommended for applications where alloy 825 does not have adequate yield or tensile strength for service in the production of oil and gas, such as valve bodies, hanger bars, flow lines, casing, and other tools and equipment.

Pyromet 860 and *Refractaloy 26* are high-temperature precipitation-hardenable alloys with lower nickel content than Inconel alloy X-750 but with additions of cobalt and molybdenum. The precipitation-hardening elements are the same except the Al/Ti ratio is reversed with titanium content being greater than aluminum. Typical applications of both alloys are critical components of gas turbines, bolts, and structural members.[8]

Nickel–Iron

The nickel–iron alloys listed in Table 1 as a group have a low coefficient of expansion that remains virtually constant to a temperature below the Curie temperature for each alloy. A major application for *Nilo alloy 36* is tooling for curing composite airframe components. The thermal expansion characteristics of *Nilo alloy 42* are particularly useful for semiconductor lead frames and glass-sealing applications.

Ni-Span-C alloy 902 and *Incoloy alloys 903 and 907* are precipitation-hardenable alloys with similar thermal expansion characteristics to Nilo alloy 42 but having different constant coefficient of expansion temperature range. Alloy 902 is frequently used in precision apparatus where elastic members must maintain a constant frequency when subjected to temperature fluctuations. Alloys 903 and 907 are being used in aircraft jet engines for members requiring high-temperature strengths to 649°C (1200°F) with thermal expansion controlled to maintain low clearance.

Nickel–Chromium–Molybdenum Alloys

This group of alloys contains 45–60% Ni and was developed for severe corrosion environments. Many of these alloys also have good oxidation resistance and some have useful strength to 1093°C (2000°F).

Hastelloy alloy X is a non-age-hardenable nickel–chromium–iron–molybdenum alloy developed for high-temperature service up to 1204°C (2200°F). Typical applications are furnace hardware subjected to oxidizing, reducing, and neutral atmospheres; aircraft jet engine tail pipes; and combustion cans and afterburner components.[5,6]

Hastelloy alloy C is a mildly age-hardenable alloy similar in composition to alloy X except nearly all the iron is replaced with molybdenum and nickel. It is highly resistant to strongly oxidizing acids, salts, and chlorine. It has good high-temperature strength. Typical applications are chemical, petrochemical, and oil refinery equipment; aircraft jet engines; and heat-treating equipment.[6,7]

Hastelloy alloy C-276 is a modification of Hastelloy alloy C where the carbon and silicon content is reduced to very low levels to diminish carbide precipitation in the heat-affected zone of weldments. Alloy C-276 is non-age-hardenable and is used in the solution-treated condition. No postwelding heat treatment is necessary for chemical-process equipment. Typical applications are chemical- and petro-chemical-process equipment, aircraft jet engines, and deep sour gas wells.[6,7]

Hastelloy alloy G is a non-age-hardenable alloy similar to the composition of alloy X but with 2% copper and 2% columbium and lower carbon content. Alloy G is resistant to pitting and stress-corrosion cracking. Typical applications are paper and pulp equipment, phosphate fertilizer, and synthetic fiber processing.[6,7]

Inconel alloy 617 is a solid-solution-strengthened alloy containing cobalt that has an exceptional combination of high-temperature strength and oxidation resistance which makes alloy 617 a useful material for gas-turbine aircraft engines and other applications involving exposure to extreme temperatures, such as, steam generator tubing and pressure vessels for advanced high-temperature gas-cooled nuclear reactors.

Inconel alloy 625, like alloy 617, is a solid-solution-strengthened alloy but containing columbium instead of cobalt. This combination of elements is responsible for superior resistance to a wide range of corrosive environments of unusual severity as well as to high-temperature effects such as oxidation and carburization. The properties of alloy 625 that make it attractive for seawater applications are freedom from pitting and crevice corrosion, high corrosion fatigue strength, high tensile strength, and resistance to chloride-ion stress-corrosion cracking. Typical applications are wire rope for mooring cables; propeller blades; submarine propeller sleeves and seals; submarine snorkel tubes; aircraft ducting, exhausts, thrust-reverser, and spray bars; and power plant scrubbers, stack liners, and bellows.

MAR-M-252, Rene′ 41, Rene′ 95, and *Astroloy* are a group of age-hardenable nickel-base alloys containing 10–15% cobalt designed for highly stressed parts operating at temperatures from 871 to 982°C (1600 to 1800°F) in jet engines. MAR-M-252 and Rene′ 41 have nearly the same composition but Rene′ 41 contains more of the age-hardening elements allowing higher strengths to be obtained. Rene′ 95, of similar base composition but in addition containing 3.5% columbium and 3.5% tungsten, is used at temperatures between 371 and 649°C (700 and 1200°F). Its primary use is as disks, shaft retaining rings, and other rotating parts in aircraft engines of various types.[6–8]

Udimet 500, 520, 600, and 700 and *Unitemp 1753* are age-hardenable, nickel-base alloys having high strength at temperatures up to 982°C (1800°F). All contain a significant amount of cobalt. Applications include jet engine gas-turbine blades, combustion chambers, rotor disks, and other high-temperature components.[6–8]

Waspaloy is an age-hardenable nickel-base alloy developed to have high strength up to 760°C (1400°F) combined with oxidation resistance to 871°C (1600°F). Applications are jet engine turbine buckets and disks, air frame assemblies, missile systems, and high-temperature bolts and fasteners.[6–8]

Nickel Powder Alloys (Dispersion Strengthened)

These oxide dispersion strengthened (ODS) alloys are produced by a proprietary powder metallurgical process using thoria as the dispersoid. The mechanical properties to a large extent are determined by the processing history. The preferred thermomechanical processing results in an oriented texture with grain aspect ratios of about 3:1 to 6:1.

TD–nickel and *TD–NiCr* are dispersion-hardened nickel alloys developing useful strengths up to 1204°C (2200°F). These alloys are difficult to fusion weld without reducing the high-temperature strength. Brazing is used in the manufacture of jet engine hardware. Applications are jet engine parts, rocket nozzles, and afterburner liners.[6–8]

Nickel Powder Alloys (Mechanically Alloyed)

Inconel alloy MA 754 and *Inconel alloy MA 6000* are ODS nickel-base alloys produced by mechanical alloying.[9,10] An yttrium oxide dispersoid imparts high creep-rupture strength up to 1149°C (2100°F). MA 6000 is also age-hardenable, which increases strength at low temperatures up to 760°C (1400°F) These mechanical alloys like the thoria-strengthened alloys described are difficult to fusion weld without reducing high-temperature strength. Useful strength is obtained by brazing. MA 754 is being used as aircraft gas-turbine vanes and bands. Applications for MA 6000 are aircraft gas turbine buckets and test grips.

3 CORROSION

It is well recognized that the potential saving is very great by utilizing available and economic practices to improve corrosion prevention and control. Not only should the designer consider initial cost of materials, but he or she should also include the cost of maintenance, length of service, downtime cost, and replacement costs. This type of cost analysis can frequently show that more highly alloyed, corrosion-resistant materials are more cost effective. The National Commission on Materials Policy concluded that one of the "most obvious opportunities for material economy is control of corrosion."

Studies have shown that the total cost of corrosion is astonishing. The overall cost of corrosion in the United States was estimated by the National Bureau of Standards in 1978 and updated by Battelle scientists in 1995. According to a report released in April, metallic corrosion costs the United States about $300 billion a year. The report, released by Battelle (Columbus, Ohio) and Specialty Steel Industry of North America (SSINA, Washington, DC), claims that about one-third of the costs of corrosion ($100 billion) is avoidable and could be saved by broader use of corrosion-resistant materials and the application of best anticorrosion technology from design through maintenance.

Since becoming commercially available shortly after the turn of the century, nickel has become very important in combating corrosion. It is a major constituent in the plated coatings and claddings applied to steel, corrosion-resistant stainless steels, copper–nickel and nickel–copper alloys, high-nickel alloys, and commercially pure nickel alloys. Not only is nickel a corrosion-resistant element in its own right, but, owing to its high tolerance for alloying, it has been possible to develop many metallurgically stable, special-purpose alloys.[11]

Figure 1 shows the relationship of these alloys and the major effect of alloying elements. Alloy 600 with 15% chromium, one of the earliest of the nickel–chromium alloys, can be thought of as the base for other alloys. Chromium imparts resistance to oxidizing environments and high-temperature strength. Increasing chromium to 30%, as in alloy 690, increases resistance to stress-corrosion cracking, nitric acid, steam, and oxidizing gases. Increasing chromium to 50% increases resistance to melting sulfates and vanadates found in fuel ash. High-temperature oxidation resistance is also improved by alloying with aluminum in conjunction with high chromium (e.g., alloy 601).

Without chromium, nickel by itself is used as a corrosion-resistant material in food processing and in high-temperature caustic and gaseous chlorine or chloride environments.

Of importance for aqueous reducing acids, oxidizing chloride environments, and seawater are alloy 625 and alloy C-276, which contain 9% and 16% molybdenum, respectively, and are among the most resistant alloys currently available. Low-level titanium and aluminum additions provide γ' strengthening while retaining good corrosion resistance, as in alloy X-750. Cobalt and other alloying element additions provide jet engine materials (superalloys) that combine high-temperature strength with resistance to gaseous oxidation and sulfidation.

Another technologically important group of materials are the higher-iron alloys, which were originally developed to conserve nickel and are often regarded as intermediate in performance and cost between nickel alloys and stainless steels. The prototype, alloy 800 (Fe/33% Ni/21% Cr), is a general purpose alloy with good high-temperature strength and resistance to steam and oxidizing or carburizing gases. Alloying with molybdenum and chromium, as in alloy 825 and alloy G, improves resistance to reducing acids and localized corrosion in chlorides.

Another important category is the nickel–copper alloys. At the higher-nickel end are the Monel alloys (30–45% Cu, balance Ni) used for corrosive chemicals such as hydrofluoric acid, and severe marine environments. At the higher-copper end are the cupronickels (10–30% Ni, balance Cu), which are widely used for marine applications because of their fouling resistance.

Nickel alloys exhibit high resistance to attack under nitriding conditions (e.g., in dissociated ammonia) and in chlorine or chloride gases. Corrosion in the latter at elevated temperatures proceeds by the formation and volatilization of chloride scales, and high-nickel contents are beneficial since nickel forms one of the least volatile chlorides. Conversely, in sulfidizing environments, high-nickel alloys without chromium can exhibit attack due to the formation of a low-melting-point $Ni\text{-}Ni_3Si_2$ eutectic. However high chromium contents appear to limit this form of attack.[5]

Friend explains corrosion reactions as wet or dry:[11]

> *The term wet corrosion usually refers to all forms of corrosive attack by aqueous solutions of electrolytes, which can range from pure water (a weak electrolyte) to aqueous solutions of acids or bases or of their salts, including neutral salts. It also includes natural environments such as the atmosphere, natural waters, soils, and others, irrespective or whether the metal is in contact with a condensed film or droplets of moisture or is completely immersed. Corrosion by aqueous environments is electrochemical in nature, assuming the presence of anodic and cathodic areas on the surface of the metal even though these areas may be so small as to be indistinguishable by experimental methods and the distance between them may be only of atomic dimensions.*
>
> *The term dry corrosion implies the absence of water or an aqueous solution. It generally is applied to metal/gas or metal/vapor reactions involving gases such as oxygen, halogens, hydrogen sulfide, and sulfur vapor and even to "dry" steam at elevated temperatures. . . . High-temperature oxidation of metals has been considered to be an electrochemical phenomenon since it involves the diffusion of metal ions outward, or of reactant ions inward, through the corrosion product film, accompanied by a flow of electrons.*

The decision to use a particular alloy in a commercial application is usually based on past corrosion experience and laboratory or field testing using test spools of candidate alloys. Most often weight loss is measured to rank various alloys; however, many service failures are due to localized attack such as pitting, crevice corrosion, intergranular corrosion, and stress-corrosion cracking, which must be measured by other means.

A number of investigations have shown the effect of nickel on the different forms of corrosion. Figure 2 shows the galvanic series of many alloys in flowing seawater. This series gives an indication of the rate of corrosion between different metals or alloys when they are electrically coupled in an electrolyte. The metal close to the active end of the chart will behave as an anode and corrode, and the metal closer to the noble end will act as a cathode and be protected.

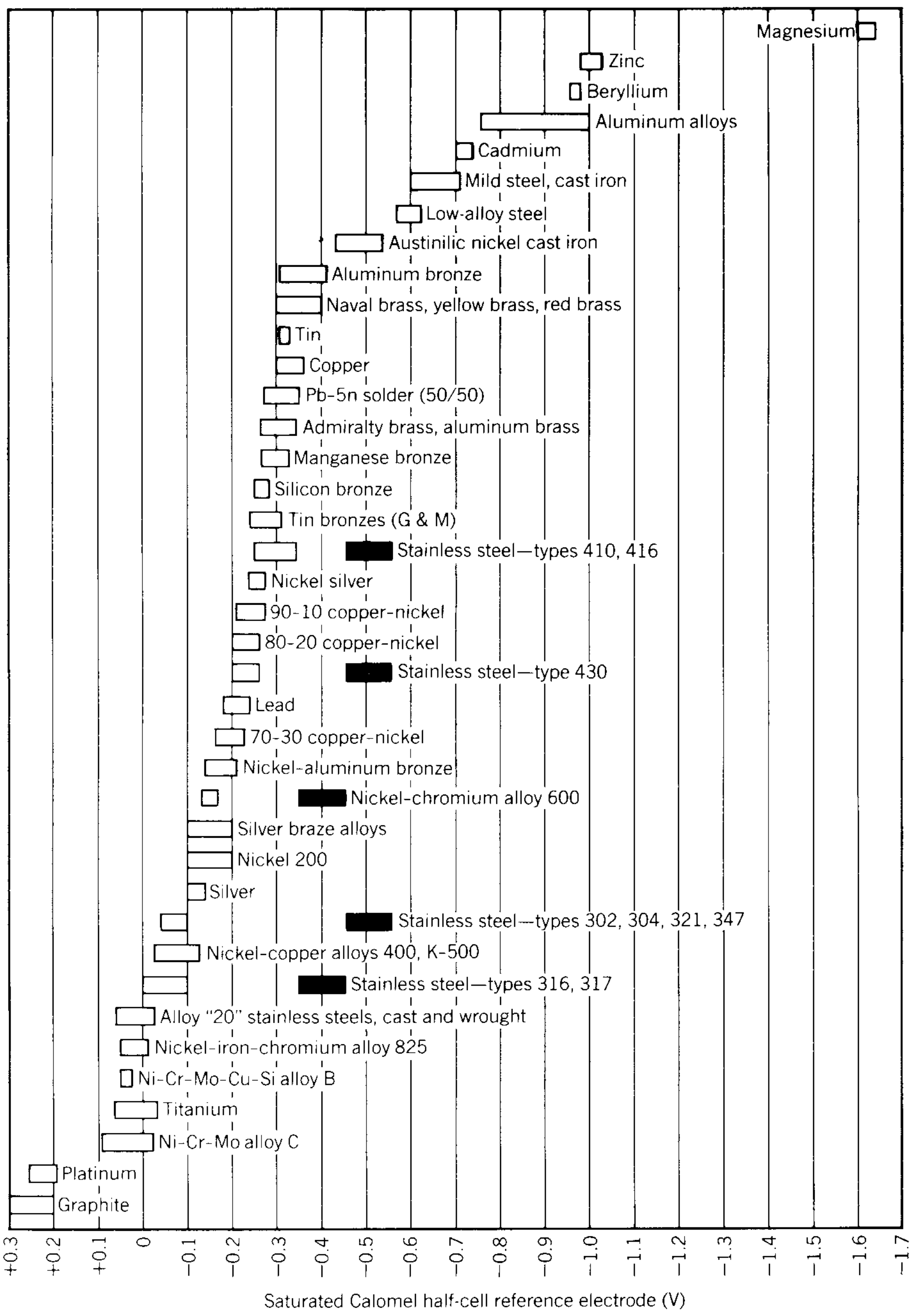

Fig. 2 Corrosion potentials in flowing seawater (8–13 ft/sec), temperature range 50–80°F. Alloys are listed in the order of the potential they exhibit in flowing seawater. Certain alloys, indicated by solid boxes, in low velocity or poorly aerated water, and at shielded areas, may become active and exhibit a potential near −0.5 V.

Increasing the nickel content will move an alloy more to the noble end of the series. There are galvanic series for other corrosive environments, and the film-forming characteristics of each material may change this series somewhat. Seawater is normally used as a rough guide to the relative positions of alloys in solution of good electrical conductivity such as mineral acids or salts.

Residual stresses from cold rolling or forming do not have any significant effect on the general corrosion rate. However, many low-nickel-containing steels are subject to stress-corrosion cracking in chloride-containing environments. Figure 3 from work by LaQue and Copson[12] shows that nickel–chromium and nickel–chromium–iron alloys containing about 45% Ni or more are immune from stress-corrosion cracking in boiling 42% magnesium chloride.[11]

When localized corrosion occurs in well-defined areas, such corrosion is commonly called *pitting attack*. This type of corrosion typically occurs when the protective film is broken or is penetrated by a chloride–iron and the film is unable to repair itself quickly. The addition of chromium and particularly molybdenum makes nickel-base alloys less susceptible to pitting attack, as shown in Fig. 4, which shows a very good relationship between critical[11] pitting temperature in a salt solution. Along with significant increases in chromium and/or

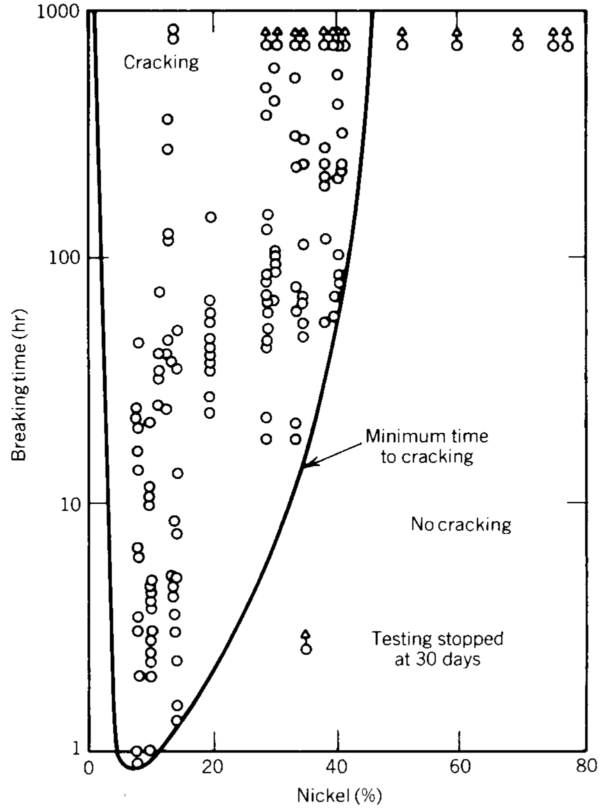

Fig. 3 Breaking time of iron–nickel–chromium wires under tensile stress in boiling 42% magnesium chloride.

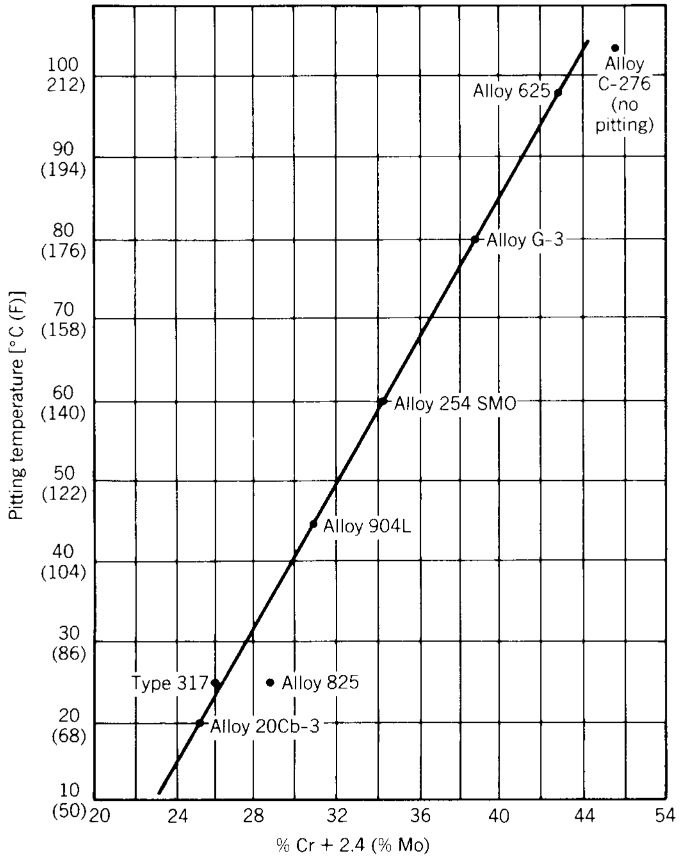

Fig. 4 Critical temperature for pitting in 4% NaCl + 1% $Fe_2(SO_4)_3$ + 0.01 *M* HCl versus composition for Fe–Ni–Cr–Mo alloys.

molybdenum, the iron content must be replaced with more nickel in wrought alloys to resist the formation of embrittling phases.[12,13]

Air *oxidation* at moderately high temperatures will form an intermediate subsurface layer between the alloy and gas quickly. Alloying of the base alloy can affect this subscale oxide and, therefore, control the rate of oxidation. At constant temperature, the resistance to oxidation is largely a function of chromium content. Early work by Eiselstein and Skinner has shown that nickel content is very beneficial under cyclic temperature conditions as shown in Fig. 5.[14]

4 FABRICATION

The excellent ductility and malleability of nickel and nickel-base alloys in the annealed condition make them adaptable to virtually all methods of cold fabrication. As other engineering properties vary within this group of alloys, formability ranges from moderately easy to difficult in relation to other materials.

4.1 Resistance to Deformation

Resistance to deformation, usually expressed in terms of hardness or yield strength, is a primary consideration in cold forming. Deformation resistance is

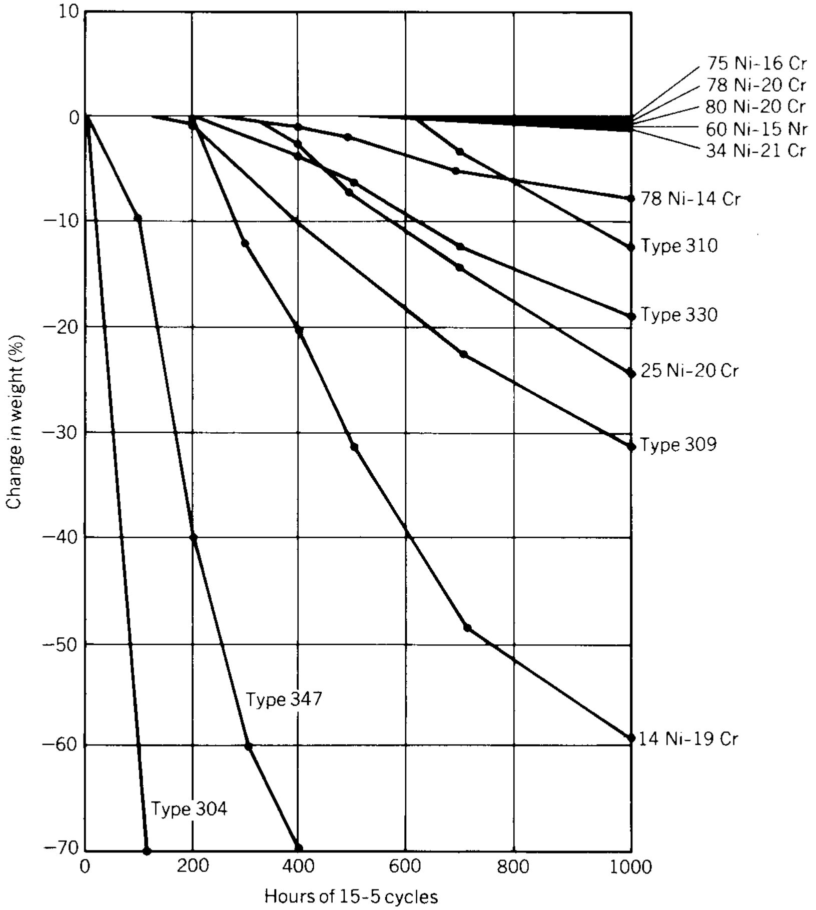

Fig. 5 Effect of nickel content on air oxidation of alloys. Each cycle consisted of 15 min at 1800°F followed by a 5-min air cooling.

moderately low for the nickel and nickel–copper systems and moderately high for the nickel–chromium and nickel–iron–chromium systems. However, when properly annealed, even the high-strength alloys have a substantial range between yield and ultimate tensile strength. This range is the plastic region of the material and all cold forming is accomplished within the limits of this region. Hence, the high-strength alloys require only stronger tooling and more powerful equipment for successful cold forming. Nominal tensile properties and hardnesses are given in Table 2.

4.2 Strain Hardening

A universal characteristic of the high-nickel alloys is that they have face-centered-cubic crystallographic structures, and, consequently, are subject to rapid strain hardening. This characteristic is used to advantage in increasing the room-temperature tensile properties and hardness of alloys that otherwise would have low mechanical strength, or in adding strength to those alloys that are hardened by a precipitation heat treatment. Because of this increased strength, large reductions can be made without rupture of the material. However, the number of reductions in a forming sequence will be limited before annealing is required, and the percentage reduction in each successive operation must be reduced.

Since strain hardening is related to the solid-solution strengthening of alloying elements, the strain-hardening rate generally increases with the complexity of the alloy. Accordingly, strain-hardening rates range from moderately low for nickel and nickel–copper alloys to moderately high for nickel–chromium and nickel–iron–chromium alloys. Similarly, the age-hardenable alloys have higher strain-hardening rates than their solid-solution equivalents. Figure 6 compares the strain-hardening rates of some nickel alloys with those of other materials as shown by the increase in hardness with increasing cold reduction.

Laboratory tests have indicated that the shear strength of the high-nickel alloys in double shear averages about 65% of the ultimate tensile strength (see Table 4). These values, however, were obtained under essentially static conditions using laboratory testing equipment having sharp edges and controlled clearances. Shear loads for well-maintained production equipment can be found in Table 5. These data were developed on a power shear having a 31 mm/m (⅜ in./ft) rake.

5 HEAT TREATMENT

High-nickel alloys are subject to surface oxidation unless heating is performed in a protective atmosphere or under vacuum. A protective atmosphere can be provided either by controlling the ratio of fuel and air to minimize oxidation or by surrounding the metal being heated with a prepared atmosphere.

Monel alloy 400, Nickel 200, and similar alloys will remain bright and free from discoloration when heated and cooled in a reducing atmosphere formed by the products of combustion. The alloys that contain chromium, aluminum, or

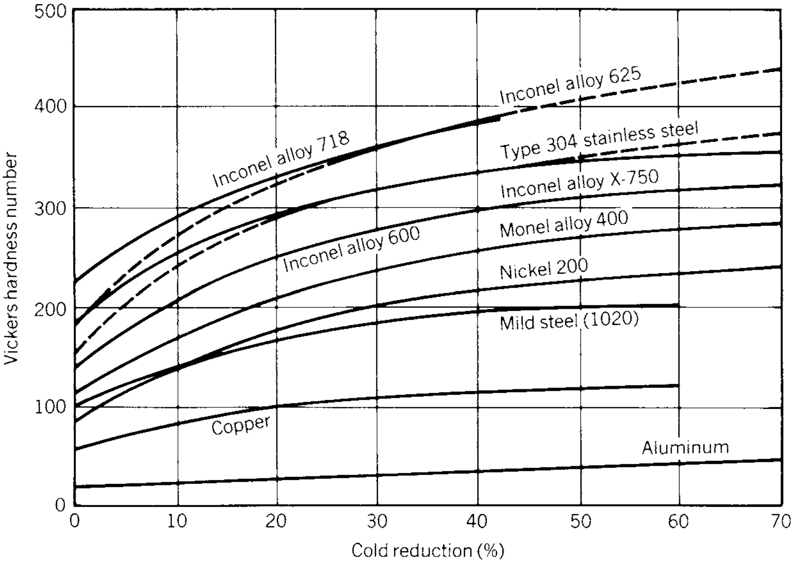

Fig. 6 Effect of cold work on hardness.

Table 4 Strength in Double Shear of Nickel and Nickel Alloys

Alloy	Condition	Shear Strength (ksi)[a]	Tensile Strength (ksi)	Hardness
Nickel 200	Annealed	52	68	46 Rb
	Half-hard	58	79	84 Rb
	Full-hard	75	121	100 Rb
Monel alloy 400	Hot-rolled, annealed	48	73	65 Rb
	Cold-rolled, annealed	49	76	60 Rb
Inconel alloy 600	Annealed	60	85	71 Rb
	Half-hard	66	98	98 Rb
	Full-Hard	82	152	31 Rc
Inconel alloy X-750	Age-hardened[b]	112	171	36 Re

[a] MPa = ksi × 6.895.
[b] Mill-annealed and aged 1300°F (750°C)/20 hr.

titanium form thin oxide films in the same atmosphere and, therefore, require prepared atmospheres to maintain bright surfaces.

Regardless of the type of atmosphere used, it must be free of sulfur. Exposure of nickel alloys to sulfur-containing atmospheres at high temperatures can cause severe sulfidation damage.

The atmosphere of concern is that in the immediate vicinity of the work, that is, the combustion gases that actually contact the surface of the metal. The true condition of the atmosphere is determined by analyzing gas samples taken at various points about the metal surface.

Furnace atmospheres can be checked for excessive sulfur by heating a small test piece of the material, for example, 13 mm (½ in.) diameter rod or 13 mm × 25 mm (½ in. × 1 in.) flat bar, to the required temperature and holding it at temperature for 10–15 min. The piece is then air cooled or water quenched and bent through 180° flat on itself. If heating conditions are correct, there will be no evidence of cracking.

Table 5 Shear Load for Power Shearing of 6.35-mm (0.250-in.) Gauge Annealed Nickel Alloys at 31 mm/m ($\frac{3}{8}$ in./ft) Rake as Compared with Mild Steel

Alloy	Tensile Strength (ksi)[a]	Hardness (Rb)	Shear Load (lb)[b]	Shear Load in Percent of Same Gauge of Mild Steel
Nickel 200	60	60	61,000	200
Monel alloy 400	77	75	66,000	210
Inconel alloy 600	92	79	51,000	160
Inconel alloy 625	124	95	55,000	180
Inconel alloy 718	121	98	50,000	160
Inconel alloy X-750	111	88	57,000	180
Mild steel	50	60	31,000	100

[a] MPa = ksi × 6.895.
[b] kg = lb × 0.4536.

5.1 Reducing Atmosphere

The most common protective atmosphere used in heating the nickel alloys is that provided by controlling the ratio between the fuel and air supplied to the burners. A suitable reducing condition can be obtained by using a slight excess of fuel so that the products of combustion contain at least 4%, preferably 6%, of carbon monoxide plus hydrogen. The atmosphere should not be permitted to alternate from reducing to oxidizing; only a slight excess of fuel over air is needed.

It is important that combustion take place before the mixture of fuel and air comes into contact with the work, otherwise the metal may be embrittled. To ensure proper combustion, ample space should be provided to burn the fuel completely before the hot gases contact the work. Direct impingement of the flame can cause cracking.

5.2 Prepared Atmosphere

Various prepared atmospheres can be introduced into the heating and cooling chambers of furnaces to prevent oxidation of nickel alloys. Although these atmospheres can be added to the products of combustion in a directly fired furnace, they are more commonly used with indirectly heated equipment. Prepared protective atmospheres suitable for use with the nickel alloys include dried hydrogen, dried nitrogen, dried argon or any other inert gas, dissociated ammonia, and cracked or partially reacted natural gas. For the protection of pure nickel and nickel–copper alloys, cracked natural gas should be limited to a dew point of −1 to 4°C (30 to 40°F).

Figure 7 indicates that at a temperature of 1093°C (2000°F), a hydrogen dew point of less than −30°C (−20°F) is required to reduce chromium oxide to chromium; at 815°C (1500°F) the dew point must be below −50°C (−60°F). The values were derived from the thermodynamic relationships of pure metals with their oxides at equilibrium, and should be used only as a guide to the behavior of complex alloys under nonequilibrium conditions. However, these curves have shown a close correlation with practical experience. For example, Inconel alloy 600 and Incoloy alloy 800 are successfully bright-annealed in hydrogen having a dew point of −35 to −40°C (−30 to −40°F).

As indicated in Fig. 7, lower dew points are required as the temperature is lowered. To minimize oxidation during cooling, the chromium-containing alloys must be cooled rapidly in a protective atmosphere.

6 WELDING

Cleanliness is the single most important requirement for successful welded joints in nickel alloys. At high temperatures, nickel and its alloys are susceptible to embrittlement by sulfur, phosphorus, lead, and other low-melting-point substances. Such substances are often present in materials used in normal manufacturing/fabrication processes; some examples are grease, oil, paint, cutting fluids, marking crayons and inks, processing chemicals, machine lubricants, and temperature-indicating sticks, pellets, or lacquers. Since it is frequently impractical to avoid the use of these materials during processing and fabrication

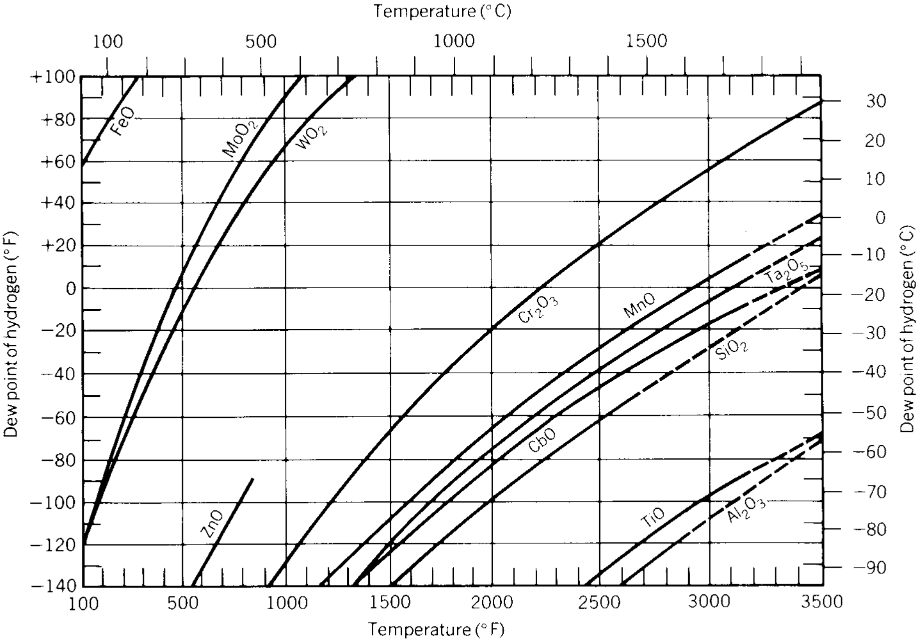

Fig. 7 Metal/metal oxide equilibria in hydrogen atmospheres.

of the alloys, it is mandatory that the metal be thoroughly cleaned prior to any welding operation or other high-temperature exposure.

Before maintenance welding is done on high-nickel alloys that have been in service, products of corrosion and other foreign materials must be removed from the vicinity of the weld. Clean, bright base metal should extend 50–75 mm (2–3 in.) from the joint on both sides of the material. This prevents embrittlement by alloying of corrosion products during the welding process. Cleaning can be done mechanically by grinding with a fine grit wheel or disk, or chemically by pickling.

7 MACHINING

Nickel and nickel-base alloys can be machined by the same techniques used for iron-base alloys. However, higher loads will be imparted to the tooling requiring heavy-duty equipment to withstand the load and coolants to dissipate the heat generated. The cutting tool edge must be maintained sharp and have the proper geometry.

8 CLOSURE

There has been a vast amount of nickel-alloy developments since the 1950 edition of *Kent's Mechanical Engineer's Handbook*. It has not been possible to give the composition and discuss each commercial alloy and, therefore, one should refer to publications like Refs. 6–8 for alloy listings, which are revised periodically to include the latest alloys available. (See Table 6 for the producer companies of some of the alloys mentioned in this chapter.)

Table 6 Registered Trademarks of Producer Company

Trademark	Owner
Duranickel	Inco family of companies
Hastelloy	Haynes International, Inc.
Incoloy	Inco family of companies
Inconel	Inco family of companies
MAR-M	Martin Marietta Corp.
Monel	Inco family of companies
Nilo	Inco family of companies
Ni–Span–C	Inco family of companies
Permanickel	Inco family of companies
Pyromet	Carpenter Technology Corp.
Rene	General Electric Co.
Rene′ 41	Allvac Metals Corp.
Udimet	Special Metals Corp.
Waspaloy	United Aircraft Corp.

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CHAPTER 8

MAGNESIUM AND ITS ALLOYS

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1 INTRODUCTION

Magnesium, with a specific gravity of only 1.74, is the lowest-density metal available for engineering use. It is produced either by electrolytic reduction of $MgCl_2$ or by chemical reduction of MgO by Si in the form of ferrosilicon. $MgCl_2$ is obtained from seawater, brine deposits, or salt lakes. MgO is obtained principally from seawater or dolomite. Because of the widespread, easy availability of magnesium ores (e.g., from the ocean), the ore supply is, in human terms, inexhaustible.

2 USES

Magnesium is used both as a structural, load-bearing material and in applications that exploit its chemical and metallurgical properties.

2.1 Nonstructural Applications

Because of its high place in the electromotive series, magnesium is used as a sacrificial anode to protect steel from corrosion; some examples are the protection of buried pipelines and the prolongation of the life of household hot-water tanks. Alloys used for this purpose are produced by permanent-mold castings and by extrusion.

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Handbook of Materials Selection. Edited by Myer Kutz

Magnesium in powder form is added to gray cast iron to produce ductile, or nodular, iron, an alloy that has many of the producibility advantages of cast iron but is ductile and strong.

A significant use for magnesium powder is its addition to the iron tapped from blast furnaces to remove sulfur prior to converting to steel, thereby increasing the efficiency of the blast furnace and improving the toughness of the steel.

Magnesium powder is also used to produce the Grignard reagent, an organic intermediate used in turn to produce fine chemicals and pharmaceuticals.

Magnesium sheet and extrusions are used to produce photoengravings.

Magnesium in ingot form is one of the principal alloying additions to aluminum, imparting improved strength and corrosion resistance to that metal.

2.2 Structural Applications

Magnesium structures are made from sand, permanent-mold, investment, and die casting, and from sheet, plate, extrusions, and forgings. The base forms produced in these ways are fabricated into finished products by machining, forming, and joining. Finishing for protective or decorative purposes is by chemical-conversion coatings, painting, or electroplating.

The most rapidly growing method of producing structural parts is die casting. This method is frequently the most economical to produce a given part and is especially effective in producing parts with very thin sections. A stimulus for the recent very high growth rate has been the development of a high-purity corrosion-resistant alloy that makes unnecessary the protective finishing of many parts. See alloy AZ91D in Table 1. Die castings are produced by cold chamber,

Table 1 Magnesium Alloys in Common Use

ASTM Designation	Ag	Al	Fe max	Mn	Ni max	Rare Earth	Si	Zn	Zr	Forms
AM50A		4.9	0.004	0.32	0.002			0.22		DC
AM60B		6.0	0.005	0.42	0.002			0.22max		DC
AS41B		4.2	0.0035	0.52	0.002		1.0	0.12		DC
AZ31B		3	0.005	0.6	0.005			1		S, P, F, E
AZ61A		6.5	0.005	0.33	0.005			0.9		F, E
AZ80A		8.5	0.005	0.31	0.005			0.5		F, E
AZ81A		7.6		0.24				0.7		SC, PM, IC
AZ91D		9	0.005	0.33	0.002			0.7		DC
AZ91E		9	0.005	0.26	0.0010			0.7		SC, PM
EZ33A						3.2		2.5	0.7	SC, PM
K1A									0.7	SC, PM
M1A				1.6						E
QE22A	2.5					2.2			0.7	S, PM, IC
WE43A			0.01	0.15	0.005	A		0.20	0.7	S, PM, IC
WE54A				0.15	0.005	B			0.7	S, PM, IC
ZE41A				0.15		1.2		4.2	0.7	S, PM, IC
ZE63A						2.6		5.8	0.7	S, PM, IC
ZK40A								4	0.7	E
ZK60A								5.5	0.7	F, E

A = 4 Yttrium; 3 RE

B = 5.1 Yttrium; 4 R.E.

DC = die casting; E = extrusion; F = forging; IC = investment casting; P = plate; PM = permanent mold; S = sheet; SC = sand casting by hot chamber, and by a recently developed method analogous to the injection molding of plastic parts. The latter technique, known as Thixomolding,[1–4] uses a machine that advances the alloy in a semisolid state by means of a screw and then injects an accumulated amount into the die. The melting step is eliminated, production rates are at least as high as for hot-chamber die casting, and metal quality is superior to that produced by either cold- or hot-chamber die casting. Two major fields dominate the die-casting markets: automotive (e.g., housings, brake pedals, transmissions, instrument panels) and computers (e.g., housings, disc readers).

Those properties mainly significant for structural applications are density (automotive and aerospace vehicle parts; portable tools such as chain saws; containers such as for computers, cameras, briefcases; sports equipment such as catcher's masks, archery bows); high damping capacity (antivibration platforms for electronic equipment; walls for sound attenuation); excellent machinability (jigs and fixtures for manufacturing processes); high corrosion-resistance in an alkaline environment (cement tools).

3 ALLOYS AND PROPERTIES

Many alloys have been developed to provide a range of properties and characteristics to meet the needs of a wide variety of applications. The most frequently used are given in Table 1. There are two major classes—one containing aluminum as the principal alloying ingredient, the other containing zirconium. Those containing aluminum are strong and ductile, and have excellent resistance to atmospheric corrosion. Since zirconium is a potent grain refiner for magnesium alloys but is incompatible with the presence of aluminum in magnesium, it is added to all alloys not containing aluminum. Within this class, those alloys containing rare earth or yttrium are especially suited to applications at temperatures ranging to as high as 300°C. Those not containing rare-earth or yttrium have zinc as a principal alloying element and are strong, ductile, and tough.

Recently, the high-purity casting alloys, AZ91E for sand and permanent mold castings and AZ91D, AM60B, AM50A, and AS41B for die castings, have been developed. The high-purity die casting alloys are superior in corrosion resistance to the commonly used aluminum die casting alloy. These alloys have been largely responsible for the large expansion in magnesium automotive applications.

3.1 Mechanical Properties of Castings

Magnesium castings are produced in sand, permanent, investment, pressure die-casting molds.

Castings produced in sand molds range in size from a few pounds to a few thousand pounds and can be very simple to extremely complex in shape. If production runs are large enough to justify higher tooling costs, then permanent instead of sand molds are used. The use of low pressure to fill a permanent mold is a low-cost method that is also used. Investment casting is a specialized technique that permits the casting of very thin and intricate sections with excellent surface and high mechanical properties. Die casting is a process for the production of castings with good dimensional tolerances, good surface, and acceptable properties at quite low cost.

Mechanical properties of cast alloys are given in Table 2.

3.2 Mechanical Properties of Wrought Products

Wrought products are produced as forgings, extrusions, sheet, and plate. Mechanical properties are given in Table 3.

3.3 Physical Properties

A selection of physical properties of pure magnesium is given in Table 4. Most of these are insensitive to alloy addition, but melting point, density, and electrical resistivity vary enough that these properties are listed for alloys in Table 5.

4 FABRICATION

4.1 Machining

Magnesium is the easiest of all metals to machine: it requires only low power and produces clean, broken chips, resulting in good surfaces even with heavy cuts.

4.2 Joining

All standard methods of joining can be used, including welding, riveting, brazing, and adhesive bonding.

Welding is by inert-gas-shielded processes using either helium or argon, and either MIG or TIG. Alloys containing more than 1.5% aluminum should be stress-relieved after welding in order to prevent stress-corrosion cracking due to

Table 2 Typical Mechanical Properties for Castings

Alloy	Temper	Tensile Strength (MPa)	Yield Strength (MPa)	Elongation in 2 in. (%)
Sand and Permanent Mold Castings				
AZ81A	T4	276	85	15
AZ91E	F	165	95	3
	T4	275	85	14
	T6	275	195	6
EZ33A	T5	160	105	3
K1A	F	185	51	20
QE22A	T6	275	205	4
WE43A	T6	235	190	4
WE54A	T6	270	195	4
ZE63A	T6	295	190	7
Investment Castings				
AZ81A	T4	275	100	12
AZ91E	F	165	100	2
	T4	275	100	12
	T5	180	100	3
	T7	275	140	5
EZ33A	T5	255	110	4
K1A	F	175	60	20
QE22A	T6	260	185	4
Die Castings				
AM50A	F	200	110	10
AM60B	F	220	130	8
AS41B	F	210	140	6
AZ91D	F	230	160	3

Table 3 Typical Mechanical Properties of Wrought Products

Alloy	Temper	Tensile Strength (MPa)	Yield Strength (MPa)		Elongation in 2 in. (%)
			Tensile	Compressive	
Sheet and Plate					
AZ31B	O	255	150	110	21
	H24	290	220	180	15
Extrusions					
AZ31B	F	260	200	95	15
AZ61A	F	310	230	130	16
AZ80A	F	340	250	140	11
	T5	380	275	240	7
M1A	F	255	180	125	12
ZK40A	T5	275	255	140	4
ZK60A	F	340	250	185	14
	T5	365	305	250	11
Forgings					
AZ31B	F	260	195	85	9
AZ61A	F	195	180	115	12
AZ80A	F	315	215	170	8
	T5	345	235	195	6
	T6	345	250	185	5
ZK60A	T5	305	205	195	16
	T6	325	270	170	11

residual stresses associated with the weld joint. Rivets for magnesium are of aluminum rather than magnesium. Galvanic attack is minimized or eliminated by using aluminum rivets made of an alloy high in magnesium, such as 5056. Brazing is used, but not extensively, since it can be done only on alloys with a high melting point, such as AZ31B or K1A. Adhesive bonding is straightforward, and no special problems related to magnesium are encountered.

4.3 Forming

Magnesium alloys are formed by all the usual techniques, such as deep drawing, bending, spinning, rubber forming, stretch forming, and dimpling.

In general, it is preferable to form magnesium in the temperature range of 150–300°C. While this requires more elaborate tooling, there is some compen-

Table 4 Physical Properties of Pure Magnesium

Density	1.718 g/cm^3 (Ref. 5)
Melting point	650°C (Ref. 6)
Boiling point	1107°C (Ref. 6)
Thermal expansion	25.2×10^{-6}/K (Ref. 7)
Specific heat	1.025 kJ/kg·K at 20°C (Ref. 8)
Latent heat of fusion	360–377 kJ/kg (Ref. 8)
Latent heat of sublimation	6113–6238 kJ/kg Ref. 6)
Latent heat of vaporization	5150–5400 kJ/kg (Ref. 6)
Heat of combustion	25,020 kJ/kg (Ref. 10)
Electrical resistivity	4.45 ohm meter $\times 10^{-8}$
Crystal structure	Close-packed hexagonal: a + 0.32087 nm; c = 0.5209 nm; c/a = 1.6236 (Ref. 9)
Young's modulus	45 Gpa
Modulus of rigidity	16.5 Gpa
Poisson's ratio	0.35

Table 5 Physical Properties of Alloys[10]

Alloy	Density (g/cm^3)	Melting Point (°C) Liquidus	Melting Point (°C) Solidus	Electrical Resistivity (ohm-metres × 10^{-8})
AM60B	1.79	615	540	
AS41B	1.77	620	565	13.0
AZ31B	1.77	632	605	9.2
AZ61A	1.8	620	525	12.5
AZ80A	1.8	610	490	15.6
AZ81A	1.80	610	490	13.0
AZ91D	1.81	595	470	17.0
EZ33A	1.83	645	545	7.0
K1A	1.74	649	648	5.7
M1A	1.76	649	648	5.4
QE22A	1.81	645	545	6.8
ZK60A	1.83	635	520	5.7

sation in the ability to produce deeper draws (thus fewer tools) and in the elimination or minimizing of springback. Hydraulic rather than mechanical presses are preferred.

5 CORROSION AND FINISHING

Magnesium is highly resistant to alkalies and to chromic and hydrofluoric acids. In these environments, no protection is usually necessary. On the other hand, magnesium is less resistant to other acidic or salt-laden environments. While most magnesium alloys can be exposed without protection to dry atmosphere, it is generally desirable to provide a protective finish.

Magnesium is anodic to any other structural metal and will be preferentially attacked in the presence of an electrolyte. Therefore, galvanic contact must be avoided by separating magnesium from other metals by the use of films and tapes. These precautions do not apply in the case of 5056 aluminum alloy, since the galvanic attack in this case is minimal.

Because magnesium is not resistant to acid attack, standing water (which will become acidic by absorption of CO_2 from the atmosphere) must be avoided by providing drain holes.

5.1 Chemical-Conversion Coatings

There are a large number of chemical-conversion processes based on chromates, fluorides, or phosphates. These are simple to apply and provide good protection themselves, in addition to being a good paint base.

5.2 Anodic Coatings

There are a number of good anodic coatings that offer excellent corrosion protection and also provide a good paint base.

5.3 Painting

If a good chemical-conversion or anodic coating is present, any paint will provide protection. Best protection results from the use of baked, alkaline-resistant paints.

5.4 Electroplating

Once a zinc coating is deposited chemically, followed by a copper strike, standard electroplating procedures can be applied to magnesium to give decorative and protective finishes.

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CHAPTER 9

CORROSION AND OXIDATION OF MAGNESIUM ALLOYS

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1 INTRODUCTION

The lightweight concept followed recently by the transportation industry made Mg alloys a highly interesting choice for many structural applications. Apart from the high strength-to-weight ratio, Mg alloys exhibit excellent die castability, superior machinability, good ductility, and damping capacity. However, the use of these alloys is far behind that of Al alloys, mainly due to their poor corrosion resistance. Although in rural and some mild industrial environments Mg alloys perform as good as Al and better than mild steel, in wet and salt-containing environments they frequently suffer severe damage. Further detrimental factors to the corrosion resistance of Mg alloys are galvanic coupling, high content of impurities (e.g., heavy metals or fluxes), surface contamination, inadequate surface protection schemes, and poor design. Even in dry environments Mg alloys oxidize easily at high temperatures of application.

The major alloying elements added to Mg in order to obtain the strong, light alloys needed for structural applications are aluminum, manganese, zinc, silicon, zirconium, rare earths (RE), and silver. Two main categories of alloys can be distinguished according to whether they contain Al or Zr. The first category

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Handbook of Materials Selection. Edited by Myer Kutz

contains 2–10% of Al; high Al contents improve corrosion resistance, however, ductility and fracture toughness are greater for lower Al contents. AM alloys contain Al and small amounts of Mn, whereas AZ, AS, and AE alloys further contain Zn, Si, and RE, respectively. Alloys from the second group contain no Al but Zr combined with alloying elements such as RE, Zn, and Ag. These alloys generally exhibit improved mechanical properties due to presence of Zr, which has a powerful grain refinement effect on magnesium. Mg alloys can be processed into casting (die, sand, and mold) or used as wrought products (extrusions, forgings, sheet, and plates); castings exceed wrought alloys and account for 85–90% of the total outcome. Detailed information on alloys and processing can be found in Ref. 1.

The prospects for the growing use of structural Mg alloys are particularly attractive but must be supported by the development of more corrosion- and heat-resistant alloys. Currently, the average amount of magnesium used in the production of automobiles is 3 kg and is expected to rise to about 6 kg in 2005.[2] Another major area in which the use of Mg is expanding is the application in electronic housings such as for computers and cell phones, where 100% recyclability, compactness arising from thin walls, and electromagnetic shielding are additional advantages. Applications for Mg alloys in automobiles are variegated and include components such as instrument and door panels, seat frames, gearbox housings, and wheels. Due to aggressive conditions such as salty road splash, high corrosion resistance is required for exterior components. The use of Mg, for example, in engine and transmission components requires additional resistance to higher temperatures. A wider exploitation of the large potential of structural Mg alloys will ultimately depend on a correct use of these alloys in oxidative and corrosive environments.

2 OXIDATION

While aqueous corrosion is recognized to be a major problem of Mg alloys, oxidation at elevated temperature is often ignored as a shortcoming for alloy application and is not very widely studied. Magnesium has a high affinity to oxygen and in its presence it rapidly reacts according to:

$$\mathrm{Mg} + \tfrac{1}{2}\mathrm{O_2} \rightarrow \mathrm{MgO} \qquad (1)$$

MgO has a face-centered-cubic (fcc) crystal structure with $a = 0.42$ nm. It is a cubic close-packing of O ions with metal ions in all octahedral sites. The free energy change that accompanies the formation of the oxide is $\Delta G_f = -1138$ kJ/mol O_2 at 25°C[3]; more negative than for most metals with only few exceptions such as Be, Ca, and some rare earths. Therefore, the resistance of Mg and its alloys to oxidation directly depends on the protective properties of the oxide film that forms first.

2.1 Ambient Temperature

Despite the very negative free energy of formation of MgO, magnesium exhibits good oxidation resistance in dry air at normal ambient temperature due to the formation of a protective oxide film. Unfortunately, the presence of water in the environment (humid air or aqueous environments) degrades the film by the formation of a less stable hydrated oxide.

An important criterion to predict the formation of a protective oxide is the Pilling–Bedworth ratio,[4] defined as the ratio of the molecular volume of the oxide to the atomic volume of the metal from which the oxide is formed. If this ratio is less than one as in the case of magnesium ($V_{\mathrm{MgO}}/V_{\mathrm{Mg}} = 0.81$), the oxide grows under internal stress leading to a discontinuous, porous film possessing low protective properties. To explain the good stability of magnesium at low temperatures, it was suggested that initially an unstable structural modification of MgO grows oriented with the metal substrate, building a very thin compact layer. Above a critical oxide thickness, however, the normal cubic lattice forms and the arising stresses lead to cracking of the film.

Generally, it is recognized that the initial oxidation of Mg proceeds by: (1) oxygen chemiesorption, (2) formation of the oxide layer (nucleation and lateral growth), and (3) oxide thickening[5–8]; in some cases a “precursor” chemisorption stage before oxide formation was claimed. Initially oxygen is adsorbed on the clean metal surface and forms a chemisorbed monolayer. A rearrangement of valence electrons of the metal and the gas is then necessary for the formation of chemical bonds. In the second stage additional layers of oxide build up forming oxide islands; place exchange and surface diffusion are the important factors. The island growth was reported to be fast and linearly dependent on the oxygen exposure; the oxide thickening stage following coalescence of the oxide islands is slow and diffusion controlled.[9] The film was reported to grow by a field-assisted cation transport mechanism described by Cabrera–Mott kinetics.[10]

A recent study[11] showed that the oxide film naturally formed on pure Mg as a result of exposure to air is thin (20–50 nm) and dense. It exhibits an amorphous structure, which is probably the reason for the protective behavior. In low-humidity atmospheres, no further increase in film thickness is observed. X-ray photoelectron spectroscopy (XPS) data indicated a mixture of elemental Mg, MgO, and 50–60 wt. % $Mg(OH)_2$.

Mg–Al alloys form similar films,[12,13] which are largely enriched in Al, especially for alloys containing more than 4% Al. Figure 1 shows that there is a terminal value of about 35% Al that can be incorporated in the oxide: All alloys containing more than 4% Al produce an oxide containing 35% Al; however, if the alloy contains less than 4% Al, the Al level in the oxide never attains 35% and becomes a function of the alloyed Al. The presence of Al in the oxide film causes no chemical shift of the XPS signal for MgO, indicating that the oxide is a mixture of MgO and Al_2O_3.

The initial air-formed film on commercial Mg alloys is also dense and protective.[14] The thin film can be visualized by transmission electron microscopy (TEM) analysis on ultramicrotomed cross sections (see Fig. 2); electron diffraction reveals its amorphous structure. Since there is no need for accommodation between the lattice of matrix and oxide, less internal stresses will develop. In addition, amorphous films are in general regarded to have better protective properties than the crystalline ones.[15]

In extended atmospheric exposures of Mg and Mg alloys, the presence of humidity and acid gases as CO_2 and SO_2 alters the composition of the oxide

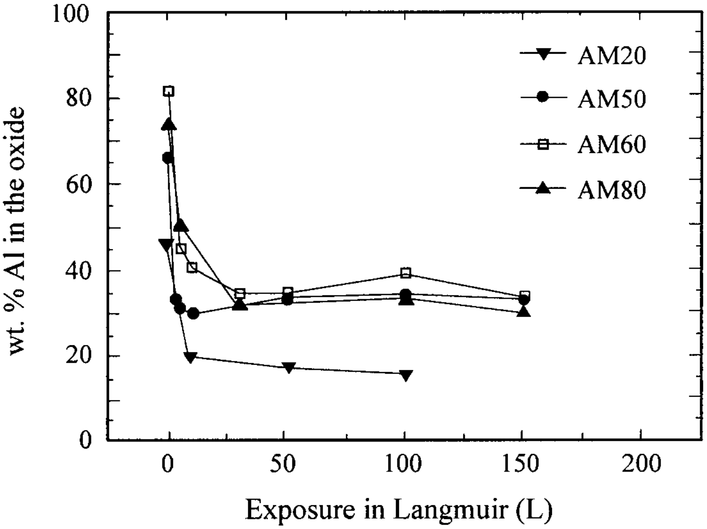

Fig. 1 Al concentration in the oxide on homogenized AM alloys (in order to dissolve the β phase) as a function of oxygen exposure.[13]

film. Hydroxides, carbonates, sulfites, or sulfates will form preferentially, thus reducing the film stability.

2.2 High Temperature

Most of the work dealing with oxidation of Mg at high temperatures is rather old.[16–20] The rate of oxidation of Mg alloys increases with the temperature obeying an Arrhenius equation. Magnesium hydroxide is not stable at high temperatures and begins to decompose above 350°C.

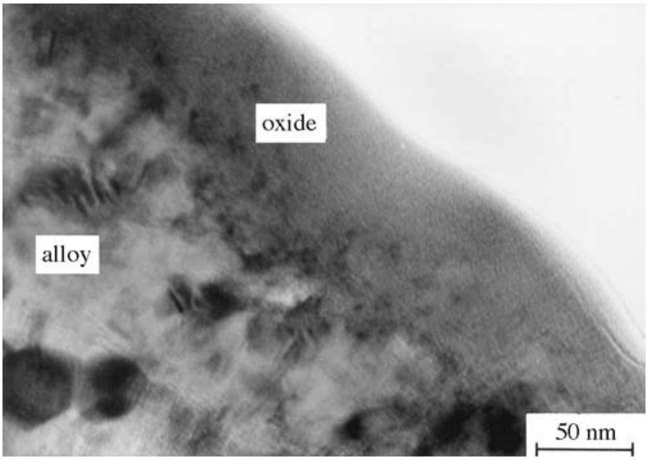

Fig. 2 TEM cross section of an oxide film formed naturally on AZ91 alloys exposed to the atmosphere.[14]

At temperatures below 450°C in dry oxygen or at 380°C in moist oxygen, the oxide film formed on magnesium is protective for considerable lengths of time;[16,18] the weight law governing the oxidation is parabolic. However, the MgO film that forms above 450°C is not protective,[16,17,19] and a linear oxidation law is observed. During this degradation of the film, a porous, white oxide grows over the surface, producing a constant oxidation rate. The micromechanism involved in this reaction is yet to be investigated. The activation energy for oxidation between 475 and 575°C was found to be about 211 kJ/mol;[17] this value may represent the true rate of the chemical reaction since in the linear regime oxygen has free access to the reaction surface. Above 600°C oxidation is extremely fast leading to ignition.

A transition in the mechanism of oxidation is also observed in commercial Mg alloys, as exemplified in Fig. 3 for high-purity AM50.[21] Two different regimes were observed in the oxidation curve of AM50 ingots at 450°C: Initially a protective oxide is formed with a parabolic kinetics ($k_p = 2 \times 10^{-13}$ $g^2/cm^4 \cdot s$) and after about 100 min a porous and less protective oxide forms with a linear kinetics ($k_l = 1.7 \times 10^{-8}$ $g/cm^2 \cdot s$). Only thin MgO films as formed at the first stages of oxidation are dense and act as a diffusion barrier for both, oxygen and metal cations. Above a certain thickness tensile stresses caused by the unfavorable Pilling–Bedworth ratio are assumed to be too high, and partial cracking of the film is likely to occur, permitting the access of oxygen to the metal surface. Additionally, if cracks allow the metal to become exposed, Mg might, in view of its appreciable vapor pressure, volatilize and react in the vapor phase with the oxygen. The initial protective period gradually shortens with the temperature since the critical thickness is more rapidly attained at higher temperature.

The effect of alloying with various metals nobler than Mg on oxidation was studied by Leontis and Rhines in the temperature range between 400 and 575°C;[17] their results are represented in Fig. 4. Most alloying elements, e.g., Al

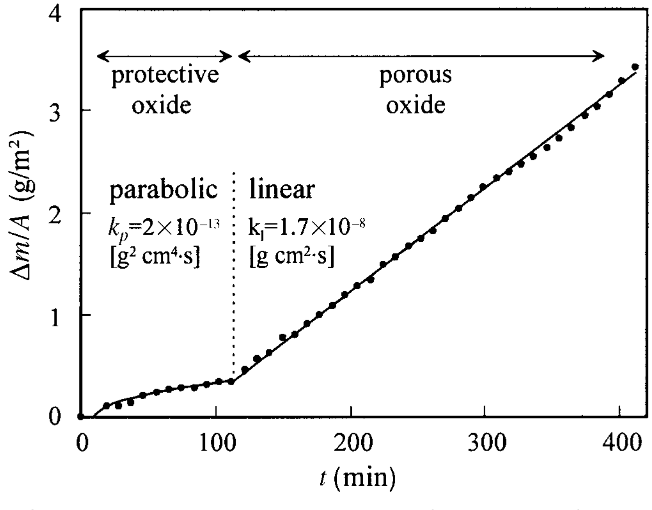

Fig. 3 Oxidation kinetics of AM50 ingots at 450°C in natural air (after Ref. 21).

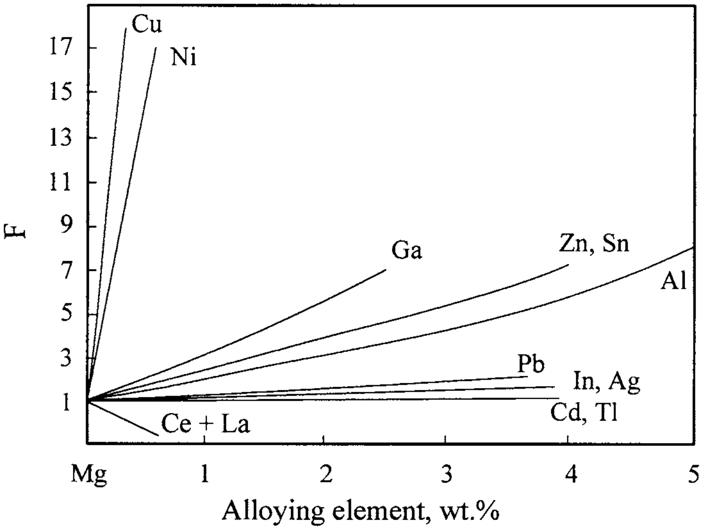

Fig. 4 Effect of alloying elements on the oxidation of Mg at ~475°C (after Ref. 22, values from Ref. 17).

and Zn, increase the rate of oxidation; only Ce and La improve the resistance of Mg to oxidation. The authors suggested that the increase in oxidation rate results from a lowering of the melting temperature by the various additions. The reactions were considered to take place at the metal/oxide interface. In contrast to Ce and La, which are enriched in the surface layers, all other alloying elements get slightly depleted in the oxide.

Ca and Be are two of the few elements that are more reactive to oxygen than Mg. They are known to have a remarkable effect on the oxidation resistance of Mg,[23] in particular when added together with Al. The addition of as little as 0.001% Be will increase the ignition temperature of Mg by as much as 200°C. Thus, small additions of Be during melting help to control Mg loss and minimize the use of fluxes. A protective BeO film forms on the surface, reducing drossing and increasing metal yield and cleanliness. These benefits carry over casting. Be provides the further advantage of precipitating Fe and Mg impurities from the melt.

Recent studies[24] performed on conventional AM and AZ alloys at 450°C confirmed the beneficial effect of RE elements predicted by Leontis and Rhines[17] and showed that Ca strongly improve oxidation performance; rapid solidification (RS) was observed to further reduce the oxidation rate. The results shown in Fig. 5 for AM50 alloys indicate that the modified alloys are very resistant to oxidation. As little as 1% of Ca is enough to reduce the oxidation rate to extremely low values, even for longer oxidation times. Rare earths (added in the form of mischmetal, Mm) are not as effective as Ca, and slightly higher contents are necessary to achieve similar performance (see Fig. 5*a*).

The oxide formed on alloys with conventional composition is thick and porous, whereas on Ca-containing alloys it is very thin so that metallic glance essentially remains after testing. The film consists of nanocrystalline MgO as

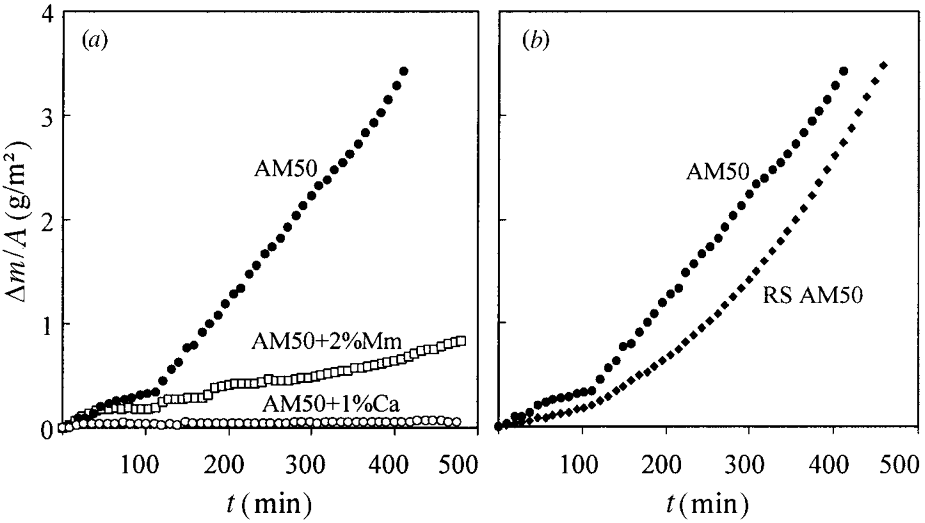

Fig. 5 Oxidation kinetics of AM50 ingots at 450°C in natural air: (*a*) effect of Ca and Mm additions and (*b*) effect of rapid solidification (after Ref. 21).

showed by X-ray defraction (XRD) and TEM cross sections (for AM50 the grain size <40 nm). In alloys containing Ca an enrichment of Ca in the MgO film in particular in its outermost part was detected by means of atomic emission spectroscopy (AES). Similar Ca enrichment was found in binary Mg–Ca alloys oxidized at 500°C, being identified as CaO.[25] Therefore, the suppression of the oxidation rate of Mg at high temperatures can be attributed to the formation of CaO, which replaces MgO near the surface and acts as a protective layer; similar explanation was given for the protection of the Mg melt by Ca additions.[26] The role of RE in the film formation is still under investigation. There is some evidence that the effect of Ca becomes detrimental at lower temperatures. For example, a recent study on AM50 alloys (same compositions as in Fig. 5) indicated that the presence of 1% Ca increases the oxidation rate at 300°C, while both unalloyed and RE-containing AM50 alloys undergo almost no oxidation at this temperature.[14] Precipitation of Al–Ca intermetallics might be responsible for this deterioration. Measurements at even lower temperatures, within the limit for application of Mg alloys, were not performed yet. Since at lower temperature kinetics becomes very sluggish, extreme long-term measurements would be necessary.

As shown in Fig. 5*b*, an additional beneficial effect can be achieved by means of RS; the improvement due to Ca and RE remains in the RS material. Until now the influence of RS on the high-temperature oxidation deserved very little attention. It seems that the advantages of RS, e.g., microstructural homogeneity, contribute toward the formation of more stable films as in the case of aqueous corrosion.

In sulfur-containing atmospheres the oxide film produced in the high-temperature range was observed to be more protective.[17] Therefore, addition of SO_2 into the atmosphere retards the oxidation of Mg at high temperatures as well as during casting. In gases containing H_2S or SO_2, magnesium alloys are among the most oxidation-resistant materials.[22] Addition of CO_2 also suppresses oxidation of solid Mg.[17]

In summary, the interaction of oxygen with Mg leads to the formation of a thin, protective oxide layer. However, temperature as well as moisture accelerate the transition to less protective films. At elevated temperatures the MgO film rapidly attains a critical thickness and starts to crack, thus allowing further oxidation. Ca and RE improve the protective properties of the MgO film and retard the transition into a porous film. Water, if present in the environment, rapidly undermines the protective film leading to a less stable, hydrated oxide and electrochemical dissolution of the metal. This process of aqueous corrosion is the subject of the next section.

3 CORROSION

In spite of the great advantages of Mg alloys, their application as engineering material is still restricted by their high susceptibility to corrosion in aqueous environments, especially in the presence of chlorides or when they contain noble metals as impurities. In this section the poor corrosion behavior of Mg alloys and the presently available methods to improve it are discussed. Over the past 30 years important improvements, which include modifications of the bulk alloy and surface, were achieved and nowadays most limitations can be successfully overcome by the correct use of Mg alloys. For further information other recent reviews are recommended.[27,28]

3.1 Electrochemical Properties

In aqueous environments magnesium dissolves electrochemically according to reaction (2) with the formation of magnesium hydroxide and hydrogen, a mechanism that is highly insensitive to the oxygen concentration.[29] If sites for easy hydrogen discharge are available, corrosion can proceed rapidly.

$$Mg + 2H_2O \rightarrow Mg(OH)_2 + H_2\uparrow \tag{2}$$

The hydroxide film has a hexagonal structure with alternating layers of Mg and hydroxide ions, thus facilitating easy basal cleavage. Cracking and curling of the film was observed, but it is not clear whether this is caused by the properties of the film or by the evolution of hydrogen gas. The Pilling–Bedworth ratio is 1.77, indicating that the film is under compression. Thus, a combination of internal stresses and the easy basal cleavage may account at least partly for the cracking and curling of the film.[30]

The standard reduction potential of Mg is −2.4 V; at higher potentials Mg corrodes rapidly. Despite the anodic polarization effect arising from the formation of $Mg(OH)_2$, the open-circuit potential in neutral aqueous solutions still falls below −1.5 V. Only at pH values higher than about 11 can a stable layer of $Mg(OH)_2$ form and provide protection against further corrosion (see Fig. 6). This protection is highly dependent on the environmental conditions, and Mg turns out to be opposite in character to Al. Mg is resistant to alkalies and poorly buffered environments where the surface pH may increase due to the initial

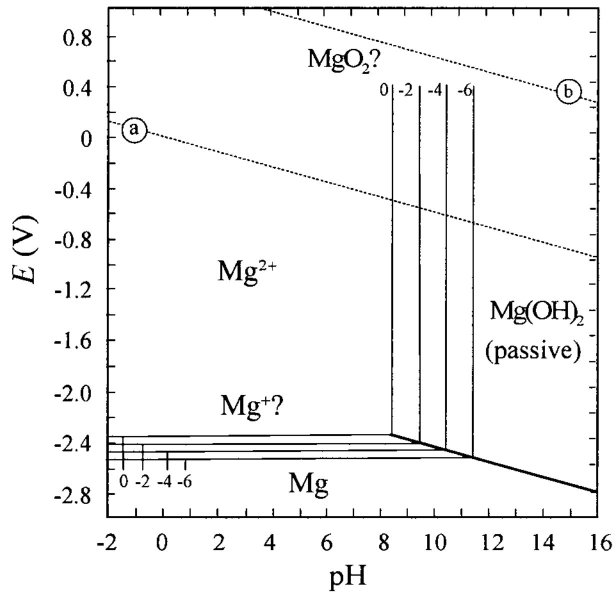

Fig. 6 Pourbaix diagram showing the equilibria for Mg–H_2O system at 25°C.[31] Lines (a) and (b) identify the hydrogen and oxygen evolution reaction, respectively.

$Mg(OH)_2$ formation but not to acids. In contrast Al is resistant to weak acids but attacked by strong alkalies.

3.2 Types of Corrosion

Mg alloys undergo both general and localized corrosion. The very electronegative potential of Mg makes these alloys very prone to galvanic corrosion. The galvanic attack can be internal due to microstructural components with more noble potential (e.g., impurities or second phases) or external if Mg is coupled with other metals. While metals with low hydrogen overvoltage (e.g., Ni, Fe, and Cu) constitute efficient cathodes for Mg and cause severe galvanic corrosion, metals that combine an active corrosion potential with a high hydrogen overvoltage (e.g., Al and Zn) are much less damaging. Internal attack can be minimized by selecting high-purity alloys. To avoid external galvanic corrosion, one of the following measures is recommended:[32] (1) Selection of galvanic compatible dissimilar metals (e.g., Al alloys 5052, 5056, and 6061; or tin, cadmium, or zinc-plated ferrous alloys). (2) Protection of Mg and dissimilar metal by suitable surface treatments; painting of the dissimilar metal or even better of both metals is recommended to avoid area effects. (3) Use of insulating washer or gasket between Mg and dissimilar metals to prevent the completion of an electrical circuit. (4) Inhibition of the galvanic cell by using chromates in the sealing compounds or primers. (5) Proper design to avoid electrolyte entrapment.

Intergranular and crevice corrosion are not likely to occur in Mg alloys because the phases at the grain boundaries are always cathodic to the grain interior and the corrosion reaction is insensitive to oxygen concentration differences.[15]

Usually, corrosion concentrates at regions adjacent to second-phase particles and boundaries. Some grains or particles might be undermined and fall out, resulting in higher mass losses than solely due to electrochemical dissolution; this partly explains the so-called negative difference effect observed during anodic polarization of Mg.[27]

As a naturally passive metal, Mg may suffer pitting corrosion. AZ91 in chloride-containing environments exhibits a variety of localized forms of corrosion, including pitting at the outset (initiation sites are few and associated with intermetallic particles), filiform corrosion at early stages of propagation, and a cellular type of attack in the terminal stage.[33–35] The occurrence of filiform corrosion on uncoated surfaces of AZ91, which is not the case for unalloyed, bare Mg,[36] suggests the presence of a more resistant air-formed oxide on the alloy.

Pure Mg is resistant to stress–corrosion cracking (SCC). However, alloying with Al and Zn promotes SCC, which increases with increasing Al content.[1] Thus, Mg–Al–Zn alloys have the greatest susceptibility to SCC. Alloys containing Zr are essentially free from the phenomenon. SCC in Mg alloys[15,37,38] is usually transgranular occurring along twin boundaries or various crystallographic planes. There is general agreement that hydrogen embrittlement is the dominant mechanism. Since hydrogen cannot penetrate the passive film unless it is already damaged, pitting is likely to be the first step on SCC. Therefore, solutions that are nonactive to Mg (such as diluted alkalies, concentrated HF, and chromic acid) or in which general corrosion predominates do not induce SCC. High-humidity content, dissolved oxygen, and seawater accelerate SCC; heavy metal content does not seem to influence it. SCC was studied mainly in Mg–Al systems and information is still lacking for other Mg alloys, in particular for new emerging alloys, e.g., glassy alloys or those containing RE.

3.3 Environment and Surface Film

Owing to the very active electrochemical character of Mg, the corrosion behavior of its alloys reflects the protective qualities of the surface film in various environments. As discussed in Section 2.1, in natural environments with low humidity a stable MgO film [or a mixture of MgO and $Mg(OH)_2$] forms on magnesium and protection is essentially perfect. Atmospheric corrosion is a threat only in the presence of humidity that leads to condensed water. The corrosion rate of magnesium during exposure to humid air is less than 10^{-5} mm/yr in the absence of condensation, while it becomes greater than 1.5×10^{-2} mm/yr when water condenses on the surface.[39] Hence, for indoor service Mg alloys serve well without any further protection.

Although stable and dense, the air-formed film is permeable to water and soluble Mg species. Exposure to humid air rapidly causes hydration of the oxide and formation of thicker films (e.g., 100–150 nm after 4 days) with a duplex structure.[11,12] The outer layer of the film (20–40 nm) is amorphous and similar to the film formed in dry air (see Fig. 2), but an additional hydrated layer is formed adjacent to the metal. The hydrated layer reduces the passivity of the metal surface by allowing Mg dissolution. This layer is quite sensitive to electron irradiation and develops a cellular structure in the microscope; continued exposure to the electron beam causes release of water and formation of nanocrystalline fcc MgO. Immersion in water leads to the formation at the outermost surface of a third layer with plateletlike morphology, which probably grows by precipitation of Mg^{2+} or other soluble Mg species that migrate outward from the innermost layer. Figure 7 shows schematically the morphology of the layered film formed in the presence of water.

Films formed in water on Mg alloys are sensitive to temperature, agitation, and contaminants.[32] The corrosion rate in water may increase by one or two orders of magnitude when the temperature rises from room temperature to around 100°C.

Immersion in a small volume of stagnant water allows rapid formation of the protective hydroxide film. Owing to the initial dissolution of hydroxide, the pH of the system rises until the point is reached where further dissolution is inhibited and the metal undergoes passivation. Agitation can prevent the pH to rise so that the solubility limit of $Mg(OH)_2$ is never reached and corrosion continues at the initial high rate. For example, AZ31B shows little attack in stagnant distilled water, but in constantly replenished water the corrosion rate is significant (0.18 mm/yr).

The presence of small amounts of dissolved salts, in particular chlorides and heavy metal salts, breaks down locally the protective film formed in aqueous environments. All solutions containing salts of heavy metals such as Fe, Ni, and Cu are extremely aggressive because the heavy metal and/or heavy metal basic salt can precipitate to form active cathodes on the anodic Mg surface.

Anions such as chloride, sulfate, and nitrate destroy the passivity of magnesium and cause severe damage. Saline environments such as marine atmospheres, salty road splash, etc. are always harsh environments for Mg alloys, even when protected. For this reason, the majority of corrosion data refer to salt spray test

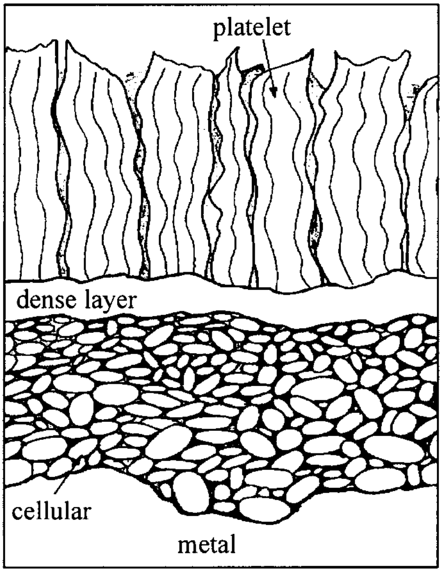

Fig. 7 Schema of the three layered oxide formed on Mg and Mg–Al alloys in water-containing environments. (Reprinted from *Corrosion Science,* **39,** J. H. Nordlien, S. Ono, N. Masuko, and K. Nisancioglu, "A TEM Investigation of Naturally Formed Oxide Films on Pure Magnesium," pp. 1397–1414. Copyright © 1997, with permission from Elsevier Science.)

and immersion in NaCl solutions operated according to the ASTM Standards B117-90 and G31-72, respectively.[40] These data must be interpreted with caution because salt based accelerate tests usually lead to higher corrosion rates than marine atmosphere exposure and show very poor correlation with rural, urban or industrial atmosphere exposure.[32]

The CO_2 in the atmosphere dissolves in water to form carbonic acid, which reacts with the $Mg(OH)_2$ film to form carbonates.[41] While these films are quite protective, they are slightly soluble in water and do not provide complete protection over extended periods of time. The same is true for other acid-producing compounds, such as SO_2 in industrial atmospheres. Exposure to wet SO_2 was suggested to accelerate the corrosion of magnesium[42] through the conversion of the more protective hydroxide and carbonate compounds to the highly soluble sulfate and sulfite, which are then eroded. If Al is present in the alloy, the effect of CO_2 is smoother due to the formation of hydrotalcite, $MgCO_3 \cdot 5Mg(OH)_2 \cdot 2Al(OH)_3 \cdot 4H_2O$, acting as a sealer for the hydroxide film. In the presence of chlorides, the accelerating effect of CO_2 on the atmospheric corrosion rate is rather weak; it becomes more important only for a low degree of salt contamination.[43]

In the presence of fluoride, the film changes from magnesium hydroxide to a very protective magnesium fluoride and protection is greatly increased, even at high temperatures. Mg is even used for constructing handling equipment for concentrated HF. Chromate also forms highly protective films on Mg, and chromic acid is the basis for many protective surface treatments.

A good rule of thumb is that environments that are basic, neutral, or contain fluorine cause little or no corrosion, while environments that are acid do cause corrosion of Mg and its alloys.

3.4 Improving Corrosion Resistance

Apart from the influence of the environment, the corrosion behavior of Mg alloys depends (similar to other metallic materials) on the presence of impurities, composition (e.g., type and amount of alloying elements) and processing methods (e.g., type of casting and heat treatment). However, the relative importance of these factors is greatly amplified for all Mg alloys because of the very negative corrosion potential of the matrix combined with the unstable nature of the surface film.

There are four main approaches that can be used to improve the corrosion behavior of Mg alloys: (1) The first approach is the production of high-purity alloys with very low impurity levels; high-purity alloys appeared in the 1980s and presently their use is standard practice. (2) Further important improvements on corrosion can be achieved by alloying with beneficial elements. This is a rather old method, however, in recent years new compositions are been studied. (3) Selective processing that optimizes the microstructure also enhances corrosion resistance; new processing techniques such as RS were shown to be very promising. (4) A further approach, which is complementary to those influencing the alloy itself, is the application of protective films or coatings.

Control of Impurities

In general, the factor with the far strongest influence on the corrosion of Mg alloys is the amount of cathodic impurities, particularly of those with low-hydrogen overvoltage. Noble impurities such as Fe, Cu, and Ni promote microgalvanic corrosion and show tolerance limits above which corrosion rate rapidly increases[44,45] (see Fig. 8). Controlling the level of contamination below these limits reduces the corrosion rate of Mg alloys containing Al by orders of magnitude, making them very competitive. For example, high-purity AZ91 shows lower salt spray corrosion rate than die cast 380 Al and cold-rolled steel[46,47] (see Fig. 8). The individual tolerance limits depend on the specific alloy composition, and a summary can be found in Ref. 27 for common high-purity alloys. For die-cast high-purity AZ91 the ASTM specification B94[48] recommends: Fe < 50 ppm, Ni < 20 ppm, Cu < 300 ppm. Mg alloys containing Zr can be considered to be of high purity because the highly reactive nature of Zr ensures that impurities present in the melt precipitate as compounds before any Zr can dissolve in the Mg matrix.[49] The tolerant limits are important for recycled alloys as well. During recycling care has to be taken to avoid contamination and secondary Mg alloys aiming structural application have to be brought within the ASTM specifications.

The detrimental effect of noble metals decreases as follows: Ni $>$ Fe $>$ Cu. Ni and Cu are usually not a problem because of their very low content in the primary production. Fe is of most concern, especially due to the risk of Fe pick up from carbon steel pots and casting molds. The solubility limit of Fe in Mg is low, and it precipitates as Al_3Fe, which acts as an effective cathode. The detrimental effect of these precipitates is reduced by small additions of Mn (up to 1%) to the melt (e.g., as $MnCl_2$).[45,50] Manganese forms several Al–Mn–Fe intermetallic particles, which can be removed after settling, and reduces the effect of the remaining Fe containing particles compared to Al_3Fe.[36,50,51] However, Mn should not be alloyed in excess of that necessary to reduce the Fe content to the appropriate level because Fe-free Al–Mn particles also exhibit

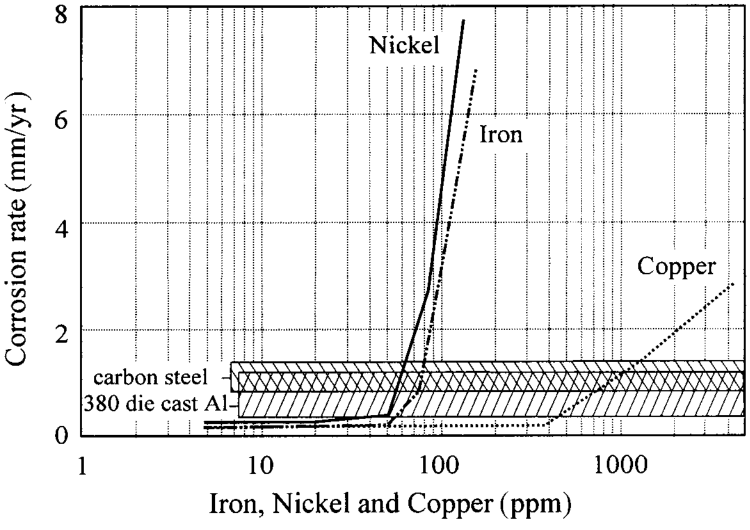

Fig. 8 Influence of Ni, Fe, and Cu impurities on the corrosion rate of die-cast AZ91 in the standard salt spray test.[47] (Reprinted with permission from SAE paper number 850417 © 1985 Society of Automotive Engineers, Inc.)

some cathodic activity, particularly the Mn-rich ones. In addition to the maximum limit for Fe, specifications for high-purity alloys include requirements on the Fe/Mn ratio (e.g., 0.032 and 0.021 for AZ91D and AM60B, respectively).[48]

Poor corrosion resistance is also associated with the presence of nonmetallic inclusions such as chlorides and oxides.[38] The development in the Mg foundry practice, which includes the use of gas mixtures (e.g., SF_6–air–CO_2), to protect the melt against excessive oxidation and the elimination of fluxes reduced substantially this concern. For a number of alloys in both cast and wrought conditions, the critical concentration of chloride for pit initiation was reported to range between 2×10^{-3} and 2×10^{-2} M NaCl.[52]

Only if impurities are controlled, other approaches such as the beneficial presence of alloying elements and the use of favorable heat treatments or RS become important.

Alloying

Keeping in mind the electrochemical properties of magnesium, it is clear that measures to enhance corrosion resistance by alloying require the use of elements with electrochemical potential close to that of magnesium (in order to avoid microgalvanic processes) and ability to help formation of the passive film. The majority of the elements conventionally alloyed to magnesium do not improve significantly corrosion resistance, e.g., addition of Zn and Si. Mn and Zr contribute significantly by reducing the effects of the impurities. The most important benefit is provided by Al, which is simultaneously the most important alloying element of Mg. RE and alkaline metals such as Ca are very attractive in regard to the electrochemical potential, and efforts are being done to develop alloys or to modify current alloys with small amounts of these elements.

Aluminum. Commercial Mg–Al alloys contain typically 2–10% of Al. In chloride environments there is a significant decrease in the corrosion rate, independent of the alloy type, as the Al is increased up to 4%. Further increase in the alloyed Al results in a smoother improvement of the corrosion resistance[51] (see Fig. 9).

Alloying with Al results in the precipitation of $Mg_{17}Al_{12}$ (β phase), in particular along grain boundaries (see Fig. 10*a*). In addition, there is some evidence for an Al gradient in the matrix, i.e., a decrease from the vicinity of the β phase toward the bulk.[33,35] $Mg_{17}Al_{12}$ precipitates and Al-rich coring zones act as a barrier against the extension of local corrosion, enhancing the corrosion resistance of Al containing alloys.

The beneficial effect of the β phase results from two effects:[33] (1) It is electrochemically nobler than the Mg matrix while having small cathodic activity as compared to Al–Mn–Fe intermetallics. (2) It is corrosion resistant over a wide pH range (4–14) by combining the passive properties of Mg in alkaline solutions with those of Al in neutral and slightly acidic media. The polarization curve of $Mg_{17}Al_{12}$ in 5% NaCl saturated with $Mg(OH)_2$ shows an anodic current plateau[54] suggesting the formation of a partially protective film. The preferential corrosion of the anodically more active bulk matrix (see Fig. 10*b*) might be the reason for the "honeycomb"-type morphology observed on corroded Al containing alloys.[50,51]

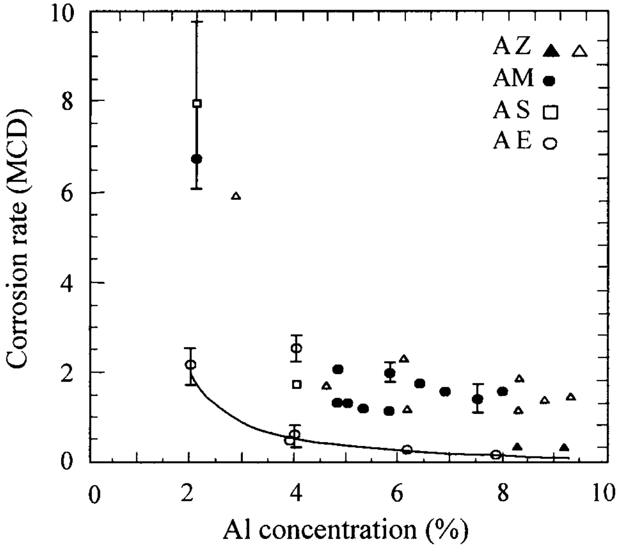

Fig. 9 Influence of Al concentration on the corrosion rate of die-cast Mg alloys during immersion in 5% NaCl.[51] (Reprinted with the permission from SAE paper number 930755 © 1993 Society of Automotive Engineers, Inc.)

Aluminum additionally improves corrosion resistance by improving the properties of the surface film. With increasing Al content all layers of the film formed on Mg–Al alloys become continuously enriched in Al_2O_3 and dehydrated, and layer thickness decreases.[12,13] Such stabilization of the film is especially advantageous for the inner layer, which is responsible for the passivity of the surface in the presence of water. The beneficial changes are significant up to 4% Al in the alloy and cause the sudden decrease in the corrosion rate observed in Fig. 9. Further alloying results in only minor changes in the oxide properties as well as in corrosion resistance. This transition relates with the alumina component in the film. Alloying up to 4% causes the oxide to enrich gradually in Al

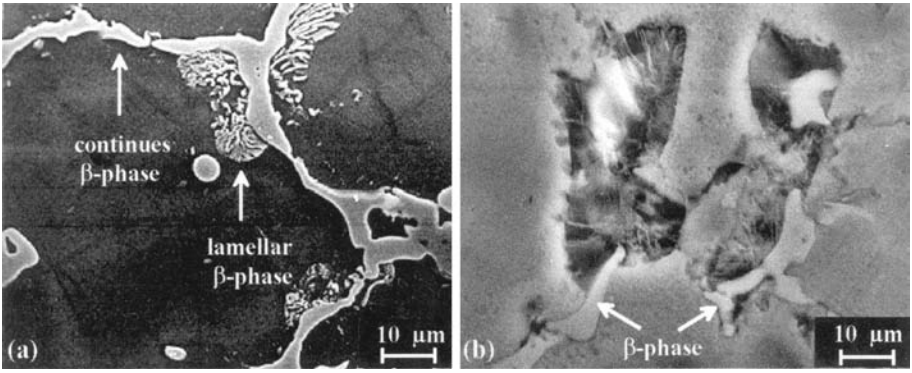

Fig. 10 AZ91 ingots: (*a*) typical microstructure and (*b*) preferential dissolution of the matrix and stability of the β phase after 24 h immersion in 5% NaCl solution.[53]

and the film properties to improve. At around 4% of alloyed Al a threshold of 35% Al in the oxide film is achieved and further alloying does not enhance corrosion resistance significantly because the amount of Al in the oxide remains unchanged. Improvements at this composition range have to be correlated with other factors such as an increase of β-phase. The Al threshold agrees well with the composition of oxides grown on the same type of alloys in ultrahigh vacuum and dry air shown in Fig. 1.[13]

Alloying with Al is an effective way to develop corrosion-resistant Mg alloys having surface films with reduced propensity for hydration and ionic conductivity. However, alloying with at least 4% Al is necessary to obtain an oxide with optimum corrosion properties. Only at this threshold the Al_2O_3 component forms a continuous passivating network, which could be a skeletal structure in the amorphous mixture of aluminum and magnesium (hydr-)oxides.[12,55]

It was also found that aluminum can have a detrimental effect on corrosion. Al was claimed to decrease the tolerance limit for Fe in an almost linear way[37,56] and in small amounts (below 8%) to produce an anodically more active Mg solid solution.[33] The negative trend in the α phase, however, reverses for high Al contents.

Rare Earth (RE) Elements. Further improvement in the corrosion resistance of Mg–Al alloys can be achieved by alloying with RE elements. RE elements further reduce the hydration of the oxide film, being a possibility to diminish the Al threshold for passivity.[57] The presence of RE promotes the enrichment of the inner oxide layer uniformly with Al_2O_3, also for Al contents in the alloy below the critical value of 4%. The formation of an oxide that is more uniformly enriched in Al agrees with the relatively small effect of RE on the oxide properties in the absence of Al.[57] An enrichment of trace amounts of RE in the oxide film was suggested by several authors but could not be definitely verified.

Another advantage of RE elements is their electrochemical potential, which being very close to that of Mg does not promote galvanic processes. Although RE show a small equilibrium solubility in the Mg matrix in the presence of Al and precipitate as Al–RE particles, these phases are electrochemically passive. Precipitation of Al_4RE was even suggested to enhance corrosion resistance due to the formation of Al coring along the grain boundaries.[51] RE elements also assist in removing Fe from the melt by settling Al–Mn–Fe–RE phases and reduce their cathodic activity.[55,58] Some RE-based intermetallics exhibit high melting temperatures and enhance creep resistance at high temperatures.

Mg–Al–Zn alloys with RE and Mn additions exhibit attractive combination of corrosion resistance, strength, and ductility. These alloys were successfully developed by Allied-Signal's Metals, both by conventionally casting and RS.[59,60] Another standard Mg alloy containing RE is the WE43 [approx. 4% Y, 3.3% Nd + hydrogen reaction equilibrium (HRE)] developed by Magnesium Elektron Ltd., which is a high-strength and corrosion-resistant alloy with long-term stability at higher temperatures. In chloride solutions AE alloys exhibit lower corrosion rates than AS, AM, and AZ alloys with similar Al content (see Fig. 9). Small additions of RE, typically 1–2%, were reported to reduce the dissolution rate of conventional AM alloys by a factor of six.[24]

Zinc. The presence of Zn improves corrosion behavior, although this effect is not very remarkable. Zinc has an appreciable solubility in the Mg matrix and the β phase. It is reported to increase the tolerance limits, although to a lesser extent than Mn,[32,44] and to make the Mg(Al) matrix more noble.[33] In alloys containing Al it was suggested to improve the stability of the oxide in the same manner as RE.[13,57] However, a related improvement in the corrosion rate of AZ alloys is not observed probably because Zn also increases the cathodic reaction rate.[54]

Silicon. Silicon is added to AS alloys where it increases the strength due to precipitation of Mg_2Si particles. Mg_2Si exhibits a corrosion potential close to that of Mg being relatively innocuous to corrosion behavior.[51] A small fraction of the alloyed Si is present in the form of Al–Mn–Fe–Si compounds and plays a similar role to that of Mn in reducing their cathodic activity.[58]

Zirconium. Mg alloys containing Zr generally exhibit good corrosion performance and are relatively insensible to Fe and Ni. Zr combines with Fe to form insoluble particles that precipitate before casting.[37] Since Zr also forms stable compounds with Al or Mn and is removed from the melt, it cannot be added to alloys containing these elements.

Calcium. The effect of Ca on the corrosion resistance of Mg is yet to be understood. Regarding the electrochemical potential, addition of Ca should result in low microgalvanic effects and reduce the anodically active area upon precipitation. This was reported for Mg–Ca–Cu splats where precipitation of Mg_2Ca neutralizes the galvanic effect from the Mg_2Cu phase. In the case of RS AZ91 alloy an addition of about 2% Ca improved the corrosion rate from 0.8 to 0.2 mm/yr.[61] However, a detrimental influence of Ca was reported for both conventional and RS binary Mg alloys.[44,62] Alloying conventional AM alloys with small amounts of Ca slightly increases the corrosion rate in NaCl.[24] Films formed on AM50+1%Ca are extremely fine nanocrystalline [identified as $Mg_6Al_2(OH)_{18} \cdot 4.5H_2O$ and $Mg(OH)_2$] and consist of at least two layers (see Fig. 11): A hydrated inner layer, which under electron irradiation develops a cellular structure and transforms into MgO, and a thin, more stable outer layer. Similar morphology is observed in films formed on Mg and Mg–Al alloys in water-containing environments (see Fig. 7). Sometimes an additional crystalline layer is observed at the interface alloy/inner layer.

Ca is very unstable in aqueous solutions and $Ca(OH)_2$ forms at pH even higher than $Mg(OH)_2$. The presence of $Ca(OH)_2$ is not necessarily expected to stabilize the Mg film due to its greater tendency to dissociate chemically in the presence of water.[31] The question whether a combination of Ca and RE is a more promising approach is now under investigation. Mechanical properties and thermal stability also benefit from the presence of Ca and RE.

Processing

For a given alloy composition corrosion properties are further influenced by the microstructure, which obviously depends on the production process. It is generally accepted that processes resulting in fine-grained, pore-free, and heat-

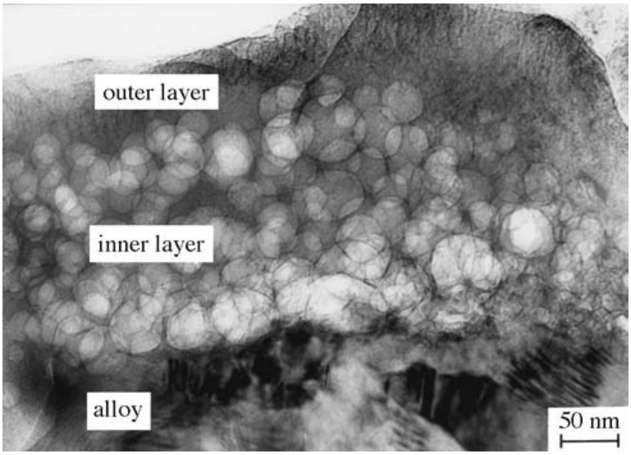

Fig. 11 TEM cross section of the film formed on AM50+1%Ca during immersion in NaCl (after Ref. 24).

treatable microstructures enhance corrosion resistance. In this connection, high cooling rates as achieved by new techniques such as RS are very useful.

Conventional Methods. Pressure die casting typically takes place at lower temperatures and solidifies in a fraction of time as compared to gravity casting, thus leading to finer microstructures. Die-cast alloys, e.g., AZ91, are generally more corrosion resistant than their mold-cast counterparts.[47] If the matrix grains are fine, a more continuous β phase forms along their boundaries, and the barrier area per material volume is larger.[58] The casting process also affects the size and distribution of cathodic phases formed when impurities are present. In AZ91 alloys the Ni tolerance was reported to be significantly lower for the gravity-cast application than for die-cast application.[47] The Fe tolerance was essentially unaffected by the cast process, being in both cases determined by the amount of Mn present. Heat treatments seem to have no influence on size and distribution of primary phases in the Al–Mn–Fe series.[33]

The corrosion resistance of Mg alloys can be further improved by heat treatments which promote the precipitation of the β phase.[33,54] In fact, the corrosion resistance of AZ91 in the artificially aged condition (T6) is superior than in the as-cast (F) or homogenized (T4) states. A reduction on the maximum pit depth is also observed in artificially aged specimens. Aging causes precipitation of secondary β phase along the grain boundaries and improves the barrier effect against corrosion. On the other hand, solution heat treatment dissolves the β phase and often leads to slightly increased corrosion rates relative to the as-cast condition.

Thixomolding is the high-speed injection molding of thixotropic, semisolid alloys. In the recent years the thixomolding process is becoming popular for production of Mg parts. In this technique fluxes are eliminated because no external melting is required and oxide formation is precluded by the use of an argon atmosphere. Thixomolded materials exhibit less porosity and nonmetallic inclusions. The corrosion rate of thixotropic-molded Mg alloys containing Al is reduced by 50% or more as compared to die-cast counterparts.[1] This reduction is attributed to an Al enrichment at the surface, due to shear thinning that results in few if any α particles at the surface, and a reduction in pinhole porosity. Die castings are more prone to porosity (due to the high velocity turbulence of molten metal entering the die) and oxide and flux inclusions.

Micropores should have a detrimental effect on the corrosion resistance of Mg parts due to the following reasons:[27] (1) Autocatalytic corrosion cells can form when electrochemical reactions inside the pore are obstructed by the corrosion products. (2) Micropores result normally from defects in the alloy, which are more active points for corrosion. (3) By increasing the real exposed surface, they increase the corrosion rate (i.e., amount of corrosion per unit area of apparent surface).

Rapid Solidification (RS). Rapid solidification introduces important microstructural changes, e.g., significant refinement of matrix and intermetallics, more homogeneous distribution of impurities, metastable extension of the solid solubility, and formation of amorphous phases, which contribute toward the formation of more protective films and the elimination of microgalvanic effects. RS can be successfully applied to Mg alloys.[63] However, one should keep in mind that the industrial feasibility of RS Mg alloys is restricted due to the limited geometry of these materials necessary to achieve high cooling rates. A common method to achieve high cooling rates during solidification is melt spinning. The melt-spun ribbons can be mechanically grounded to powders, sealed in cans, and extruded to produce bars. Another way to overcome the problem of geometry might be the application of the corrosion-resistant RS material at the surface, e.g., by laser melting/alloying.

As in conventional alloys, Al suppresses the corrosion rate of binary RS Mg alloys, in particular for large additions; however, addition of Zn, Si, and Ca enhances it.[62,64] Zinc shows a somewhat anomalous effect, causing the corrosion rate of RS magnesium to increase to a maximum at 18.6% Zn and then decrease with further additions.

RS intensifies the benefits of Al and RE in the stabilization of the passive film in two ways: It increases their solid solubility limits and provides a more homogeneous substrate where it is easier to form the film. The maximum solid solubility of Al in Mg was extended up to 24.5 or 23.4% by splat cooling or melt spinning, respectively.[65,66] In 0.001 M NaCl an anodic plateau develops at low currents, the pitting potential shifts to more noble values, and the corrosion rate decreases by two orders of magnitude when the Al in solid solution is extended from 9.6 to 23.4%.[66] Homogeneous Mg(Al) solid solutions are advantageous because of the formation of Al-stabilized film over the entire surface. Improvements in the passivation behavior in 0.01 M NaCl were also observed in several RS alloys containing RE, e.g., Mg–Zn–Zr–RE (RE = Nd, Ce, or Y), Mg–Nd, and Mg–Y.[67,68] As mentioned above, it is not clear whether RE elements become enriched in the surface film; in RS alloys both enrichment[68,69] and depletion[70] were reported.

If the films become more stable along with a more homogeneous alloy microstructure, susceptibility to pitting should be reduced. This was observed for AZ61, where RS increases the breakdown potential by about 200 mV in Cl^--

containing solutions.[62] Since pitting is assumed as a necessary precursor to SCC, RS might help to produce SCC-resistant Mg alloys.

RS was also suggested to increase the tolerance limit for Fe.[71] The ennoblement of the matrix with Al and the reduced rate of proton discharge around the refined Fe inclusions were considered to be the origin of this improvement. RS does not necessarily eliminate microgalvanic reaction between Mg matrix and heavy metal precipitates as shown by the higher corrosion rates of RS Mg–Mn alloys containing Ni in comparison with RS pure Mg.[72] However, if RS leads to a sufficiently fine partially crystallized or fully amorphous structure (where heavy metals and Mn are dispersed or even solved), the microgalvanic reaction is suppressed and the corrosion moves to a more general kind.[73,74]

RS Mg–Al–Zn alloys (around 9–21% Al and 3–7% Zn) exhibit up to one order of magnitude lower corrosion rates and better anodic polarization behavior in NaCl than the conventionally cast counterparts.[75] A several years development program on extruded RS Mg–Al–Zn alloys with, e.g., Mn and RE additions, resulted in the evaluation of alloys, e.g., EA55RS (4.8–5.2% Al + 4.7–5.0% Zn + 5.0–5.5% Nd), with better performance than the corrosion resistant AZ91.[76] Figure 12 shows that corrosion rates of RS alloys in NaCl are approaching to those of Al alloys.

Amorphous materials are well known for their improved corrosion resistance, arising from the chemically homogeneous single-phase nature without compositional fluctuations and crystal defects. Glass-forming ability can be found in many Mg systems, e.g., in Mg–Ni, Mg–Cu, Mg–Ln (Ln = lanthanide metal), Mg–Y and Mg–Ca systems often with ternary additions[63] but allows only thin glassy ribbons. Many of these new partially or fully amorphous alloys are attractive, since they attain high corrosion resistance. For example, amorphous and/or nanocrystalline Mg–Y (20–26 at.% Y) films produced by magnetron cosputter deposition perform better in 0.1 *M* NaCl than pure Mg and even the

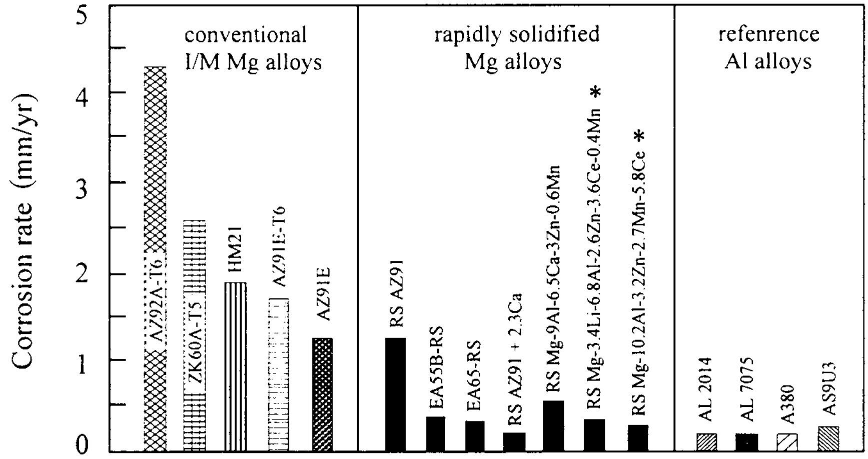

Fig. 12 Corrosion rate of light alloys obtained by weight loss measurements in 5% NaCl solution. * Data from electrochemical impedance spectroscopy. (Reprinted, by permission from Ref. 61. Copyright © 1996 by Wiley-VCH.)

WE43 alloy.[69] Recently, new perspectives were open with bulk amorphous alloys in the system Mg–TM–Ln (where TM is Ni or Cu and Ln is a lanthanide metal),[77–79] which could be used as structural materials. These glasses display an exceptionally large glass-forming ability and high thermal stability. The supercooled liquid region before crystallization is very wide (>60°C for $Mg_{65}Cu_{25}Y_{10}$ and >40°C for $Mg_{50}Ni_{30}Y_{20}$) and the temperature of crystallization increases with the solute content.[77,80] The most promising bulk amorphous alloy is $Mg_{65}Cu_{25}Y_{10,}$ which can be produced with a thickness of 4 mm by metallic mold casting and 7 mm by high-pressure die casting[77,78] and exhibits good corrosion resistance.[81,82] Bulk amorphous Mg–Ni–Nd alloys were obtained with a thickness of 3.5 mm and show higher corrosion resistance than AZ91E and EA55RS alloys in 3.5% NaCl solutions.[83]

Surface Protection

As already pointed out, the films that form on Mg, although quite protective in the absence of humidity and contaminants, do not serve perfectly in all environments. Coatings are used to supplement or replace the natural film, or as better foundation for subsequently applied paint. Direct application of a paint on Mg alloys is difficult due to the basic character of the naturally formed film, typically with a pH of 10.5. Coating can be done by chemical conversion, metal plating, anodization, or application of an organic coat.[84] Effective protection against corrosion usually requires finishing schemes that combine several of these techniques.

Traditional chemical treatments for Mg are generally based on chromate solutions, which are under increasing environmental regulation. With high-purity alloys, selected phosphate treatments can be as effective as chromates. Chemical treatments increase paintability and retard the natural alkali that forms at any point of damage on a painted Mg surface. Used alone they do not provide enough corrosion protection (among other reasons due to their solubility in water).

Any metal that can be electrodeposited can be applied successfully to Mg, but caution should be taken when using electroplating to protect components exposed to corrosive environments. This process is used to attain bright, tarnish-resistant surfaces and to improve wear resistance. In general, plating of Mg consists of a zinc immersion plating and a copper strike, followed by electroplating in standard plating baths. Copper–nickel–chromium plating systems provide good protection, even though galvanic corrosion problems would be expected when the plate is damaged.

Anodic coatings combine reasonable corrosion resistance with excellent paint base qualities and abrasion resistance. These hard ceramiclike coatings exhibit different degrees of inherent porosity. The pores must be sealed to enhance corrosion resistance. The coatings can be filled with polymers such as polytetrafluoroethylene (PTFE) to obtain special properties, e.g., lubricity. Typical anodic coating processes are HAE and Dow 17.[1] Both processes give rise to coatings with thickness in the range of 5–30 μm, consisting mainly of mixed oxides. Anodic coatings represent a special type of chemical conversion coating because some Mg is used to build up the protecting film. The Dow 17 process utilizes a chromate-based formulation. Permanganate is the key ingredient in the

HAE process, however, a chromate sealant is necessary to obtain acceptable corrosion resistance.

Tagnite[85] and Magoxid-Coat[86] are anodic spark processes carried out at higher voltages (final voltage up to about 400 V AC). These anodic coatings are more protective and not based on chromates. The Tagnite process uses a hydroxide–silicate–fluorid electrolyte and the thickness of the coating can be varied between 2 and 30 μm depending on current density and time. Magoxid-Coat is formed in a slightly alkaline electrolyte and results in a partially amorphous oxidic layer based on Mg, Al, and P. The coating consists of a thin barrier layer, a virtually nonporous intermediate ceramic layer responsible for the good corrosion resistance and a very porous outermost ceramic layer. The coating builds up to an optimal thickness of 15–25 μm. Probably, the amorphous nature of the coating contributes toward its improved corrosion behavior. Codeposition of transition metal oxides and organic materials is used to produce additional characteristics, e.g., colored coatings.

Painting of Mg parts may be used to apply a decorative finish or as a mean of protection against corrosion and tarnish. Organic coating can vary from simple oils or waxes to multipaint coatings. If the environment is severe, primers should be based in alkali-resistant vehicles as, e.g., vinyl, epoxy, polyvinyl butyral, acrylic resins, or baked phenolic resins, and contain inhibitive pigments with a slightly soluble chromate.

Following the recent developments of surface engineering, a variety of new surface modification technologies, including ion implantation, chemical vapor deposition/physical vapor deposition (CVD/PVD), and laser surface alloying/melting, can be used to develop more effective and environmental friendly coatings.[87] Hydride coatings were recently proposed as an alternative to chromate coatings.[88] A magnesium hydride or hydrogen-rich layer with thickness of 1–2 μm is created on the Mg surface by cathodic electric charging in aqueous solution. The hydride coating promotes pseudo-passivation of Mg. It can be used either as a stand-alone protective coating or as an effective paint base. Implantation of N^{2+} ions on AZ91D surfaces enhances corrosion resistance; an optimal dose of 5×10^{16} ions/cm^2 results in a corrosion rate approximately 15% of that for the unimplanted alloy.[89]

4 OUTLOOK

Whereas oxidation resistance of Mg alloys is generally considered to be sufficient, at least in the temperature range of typical application, corrosion can still be a problem. Further trends to improve corrosion properties of Mg alloys include: (1) Control of heavy metal content, (2) alloying with elements that stabilize the surface oxide, e.g., RE, and with Mn that improves corrosion behavior due to iron control effect, (3) use of RS techniques, and (4) coating and painting.

Heavy metal impurity control is the key action in the design of highly corrosion resistant Mg alloys. These impurities enhance corrosion by orders of magnitude and only their accurate control gives the indispensable basis for a successful application of all other corrosion protection measures.

In marked contrast to Al and its alloys, Mg does not form a dense protective oxide. The films naturally formed on Mg, although stable in dry environments, are rapidly undermined in the presence of water and contaminants. The usefulness of Mg alloys can be successfully increased by improving the oxide layer or by applying an appropriate coating. The first approach involves alloy development with the objective of reducing the permeability to water and improving the adhesion to the surface of the air-formed film. This can be achieved by alloying with, e.g., Al and RE, resulting in more stable oxides with reduced propensity for hydration. At least 4% of Al seems to be necessary to form a protective, self-healing film but this threshold is reduced by small additions of RE. RE elements also contribute to improve the oxidation resistance at high temperatures.

RS is a very efficient technique to improve both general and localized corrosion resistance of Mg alloys. Improvements arise from the (1) higher homogeneity of the microstructure, (2) extended solubility of passivation elements, such as Al, and (3) formation of amorphous phases. In terms of corrosion only the material at the surface needs to perform well. Thus, improvements can be obtained by producing an RS alloy surface, e.g., by laser melting/alloying. In general, amorphous materials are particularly resistant against corrosion. Recent developments in amorphous Mg alloys with high thermal stability opened up new prospects to obtain such alloys in bulk castings, especially in the Mg–Cu–Y system.

Thixomolding is an emerging technique especially attractive to produce net-shape Mg parts. Due to the reduced porosity, properties of thixomolded products are generally better than those of die castings.

Despite all these improvements, surface protection will continue to be necessary in harsh environments. New anodic coatings (e.g., Magoxid-Coat) exhibit excellent corrosion and wear resistance. These coatings are environmentally more friendly than the processes based on chromates, a known carcinogen, in the primary coating or sealing baths (e.g., Dow17 and HAE). New emerging hydride coatings may constitute attractive alternatives to the established ones.

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CHAPTER 10

SELECTION OF SUPERALLOYS FOR DESIGN

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1 INTRODUCTION

1.1 Purpose

The purpose of this chapter is to provide a reasonable understanding of superalloys so that selection of them for specific designs will be appropriate. Knowl-

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edge of superalloy types and their processing will give a potential user the ability to understand the ways in which superalloys can contribute to a design. More importantly, the knowledge provided here should enable the user to ask the important questions of superalloy providers to determine the necessary mechanical property and corrosion/environmental behavior that will influence alloy selection. There is no cookbook for superalloy selection. Proprietary alloys and/or proprietary/restricted processing lead to superalloy conditions and properties not listed in a handbook or catalog of materials. With few exceptions, critical applications will require work with one or more superalloy producers to develop an understanding of what is available and what a superalloy selector/designer can expect from a chosen superalloy.

1.2 What Are Superalloys?

Superalloys for purposes of this chapter are those nickel-, iron-nickel-, and cobalt-base corrosion-resistant alloys generally used at temperatures above around 540°C (1000°F). The iron-nickel-base superalloys are an extension of stainless steel technology and generally are wrought, i.e., formed to shape or mostly to shape by hot rolling, forging, etc. The cobalt-base and nickel-base superalloys, on the other hand, may be either wrought or cast depending on the application or the alloy composition involved. The stainless steels, nickel–chromium alloys and cobalt dental alloys that evolved into the superalloys used chromium to provide elevated temperature corrosion resistance. A Cr_2O_3 layer on the surface proved very effective in protection against oxidation. Eventually, cast superalloys for the highest temperatures were protected against oxidation by chromium and aluminum. In our opinion, superalloys must contain chromium, probably at the level of 5% (some would argue 8%) or higher.

1.3 How Are They Strengthened?

Metals are crystalline and the atoms take various crystallographic forms. Some of these forms tend to be associated with better property characteristics than other crystal structures. By introducing atoms, phases, or grain boundaries, the movement of imperfections that cause deformation to occur is inhibited. The process of modifying composition and microstructure enables materials to be strengthened.

Superalloys consist of an austenitic face-centered-cubic (fcc) crystal structure matrix phase, gamma, plus a variety of secondary phases. Important secondary phases are γ' fcc ordered $Ni_3(Al, Ti)$ and various MC, $M_{23}C_6$, M_6C, and M_7C_3 (rare) carbides in nickel- and iron-nickel-base superalloys. Carbides are the principal secondary phases in cobalt-base alloys. Also, γ'', a body-centered-tetragonal (bct) phase of ordered Ni_3Nb, a hexagonal ordered Ni_3Ti (η) phase, and the δ-orthorhombic Ni_3Nb intermetallic phase can be found in nickel- and iron-nickel–base superalloys.

It is quite important for the alloy selector to have a realistic understanding of the strengthening process in superalloys as the properties of superalloys can be modified considerably by processing to manipulate the strengthening level achieved. The superalloys derive their strength from solid-solution hardeners and secondary precipitate phases that form in the γ matrix and produce precipitation (age) hardening. Principal strengthening precipitate phases in nickel-base and iron-nickel-base superalloys are γ' and γ''. Carbides may provide limited strengthening directly (e.g., through dispersion hardening) or, more commonly, indirectly (e.g., by stabilizing grain boundaries against movement). The δ- and η-phases are useful (along with γ') in control of grain structure of wrought superalloys during processing. By controlling grain structure, strength can be significantly influenced. The extent to which the second phases contribute directly to strengthening depends on the alloy and its processing. It should be noted that improper distributions of carbides and precipitate phases can be detrimental to properties.

In addition to those elements that produce solid-solution hardening and/or promote carbide and γ' formation, other elements (e.g., boron, zirconium, hafnium) are added to enhance mechanical or chemical properties. Superalloy microstructure and property control can be complex. As many as 14 elements may be controlled in some superalloys. Some carbide- and γ'-forming elements may contribute significantly to corrosion properties as well. Tables 1 and 2 give a generalized list of the ranges of alloying elements and their effects in superalloys.

1.4 Manufacture of Superalloy Articles

Appropriate compositions of all superalloys can be forged, rolled to sheet, or otherwise formed into a variety of shapes. The more highly alloyed superalloy compositions normally are processed as small castings. Large castings are made only in the weldable superalloy IN 718. Fabricated superalloy structures can be built up by welding or brazing, but the more highly alloyed the superalloy composition (i.e., higher amount of hardening phase), the more difficult it is to weld a superalloy. Machining of superalloys is similar to but more difficult than that of machining stainless steels. In welding or machining of superalloys, the effects of the energy input (heat energy, deformation energy) on the microstructure and properties of the final superalloy product must be considered.

Iron-nickel- and nickel-base superalloys are readily available in extruded, forged, or rolled form; the higher-strength alloys generally are found only in the cast condition. Hot deformation is the preferred forming process, cold forming

Table 1 Common Ranges of Major Alloying Additions in Superalloys

	Range, (%)	
Element	Fe–Ni and Ni Base	Co Base
Cr	5–25	19–30
Mo, W	0–12	0–11
Al	0–6	0–4.5
Ti	0–6	0–4
Co	0–20	—
Ni	—	0–22
Nb	0–5	0–4
Ta	0–12	0–9
Re	0–6	0–2

Source: Metals Handbook Desk Edition, ASM International, Materials Park, OH 44073-0002, 1999, p. 395.

Table 2 Role of Alloying Elements in Superalloys

Effect[a]	Iron Base	Cobalt Base	Nickel Base
Solid-solution strengtheners	Cr, Mo	Nb, Cr, Mo, Ni, W, Ta	Co, Cr, Fe, Mo, W, Ta, Re
fcc matrix stabilizer	C, W, Ni	Ni	—
Carbide form			
MC	Ti	Ti	W, Ta, Ti, Mo, Nb, Hf
M_7C_3	—	Cr	Cr
$M_{23}C_6$	Cr	Cr	Cr, Mo, W
M_6C	Mo	Mo, W	Mo, W, Nb
Carbonitrides: M(CN)	C, N	C, N	C, N
Promotes general precipitation of carbides	P	—	—
Forms γ' $Ni_3(Al,Ti)$	Al, Ni, Ti	—	Al, Ti
Retards formation of hexagonal η (Ni_3Ti)	Al, Zr	—	—
Raises solvus temperature of γ'	—	—	Co
Hardening precipitates and/or intermetallics	Al, Ti, Nb	Al, Mo, Ti[b], W, Ta	Al, Ti, Nb
Oxidation resistance	Cr	Al, Cr	Al, Cr, Y, La, Ce
Improve hot corrosion resistance	La, Y	La, Y, Th	La, Th
Sulfidation resistance	Cr	Cr	Cr, Co, Si
Improves creep properties	B	—	B, Ta
Increases rupture strength	B	B, Zr	B[c]
Grain-boundary refiners	—	—	B, C, Zr, Hf
Facilitates working	—	Ni_3Ti	—
Retard γ' coarsening	—	—	Re

[a] Not all these effects necessarily occur in a given alloy.

[b] Hardening by precipitation of Ni_3Ti also occurs if sufficient Ni is prsent.

[c] If present in large amounts, borides are formed.

Source: Metals Handbook Desk Edition, ASM International, Materials Park, OH 44073-0002, 1999, p. 395.

usually being restricted to thin sections (sheet). Cold rolling may be used to increase short-time strength properties for applications at temperatures below the lower temperature level of about 540°C (1000°F) established in this chapter for superalloy use. Properties of superalloys generally are controlled by adjustments in chemistry (composition) and by modification of the processing (including heat treatment).

1.5 Superalloy Information

While some chemistries and properties are listed in this chapter, there is no substitute for consultation with superalloy manufacturers about the forms (cast, wrought) that can be provided and the exact chemistries available. A few producers are noted at the end of this chapter. Also, it should be understood that not all superalloys are readily available as off-the-shelf items. While literally hundreds, perhaps thousands of superalloy compositions have been evaluated since the mid-1930s, only a handful are routinely produced. Moreover, some superalloys are not available for use in all forms and sizes. Many of the highest strength alloys will only be useful as powder metal products or as castings.

Design data for superalloys are not intended to be conveyed here, but typical properties are indicated for some materials. Design properties should be obtained from internal testing if possible or from producers or other validated sources if sufficient test data is not available inhouse. Typical properties are merely a guide for comparison. Exact chemistry, section size, heat treatment, and other processing steps must be known to generate adequate property values for design.

The properties of the extraordinary range of superalloy compositions developed over the years are not normally well documented in the literature. However, since many consumers of superalloys actually use only a few alloys, within the customary user groups, data may be more plentiful for certain compositions. The extent to which such data are available to the general public is unknown. However, even if such data were disseminated widely, the alloy selector needs to be aware that processing treatments such as forging conditions, heat treatment, coatings for corrosion protection, etc. dramatically affect properties of superalloys. All data should be reconciled with the actual manufacturing specifications and processing conditions expected. Alloy selectors should work with competent metallurgical engineers to establish the validity of data intended for design as well as to specify the processing conditions that will be used for component production.

Application of design data must take into consideration the probability of components containing locally inhomogenous regions. For wrought superalloys, the probability of occurrence of these regions (which are highly detrimental to fatigue life) is dependent upon the melting method selected (see Section 5 melting for details). For cast superalloys, the degree of inhomogeneity and the likelihood of defects such as porosity is related to the alloy composition and the investment casting technique used to produce a component.

For sources of property data other than that of the producers (melters, forgers, etc.) or an alloy selector's own institution, one may refer to organizations, such as ASM International, that publish compilations of data that may form a basis for the development of design allowables for superalloys.

Standards organizations, such as the American Society for Testing and Materials (ASTM), publish information about superalloys but that information may not ordinarily contain any design data. The great versatility of superalloys in property modification is also a detriment to the universal compilation of property values. The same nominal alloy may have some composition modifications occur from one manufacturer or customer to another. Sometimes this extends from one country to another. Tweaking of the casting or forging process or the heat treatment, in addition to what seem like minor composition changes, can cause significant variations in properties. All facets of chemistry and processing need to be considered when selecting a superalloy for an application.

Nominal compositions of selected superalloys are provided in Table 3. Physical properties of a few superalloys are given in Tables 4 and 5, short-time (tensile) properties in Tables 6 and 7, and time-dependent (creep-rupture) properties in Tables 8 and 9. Selected reference publications for superalloy technical information or properties are indicated in the bibliography.

2 METALS AT HIGH TEMPERATURES

2.1 General

While material strengths at low temperatures are usually not a function of time, at high temperatures the time of load application becomes very significant for

Table 3 Nominal Compositions of Wrought Superalloys

Alloy	UNS Number	Composition (%)										
		Cr	Ni	Co	Mo	W	Nb	Ti	Al	Fe	C	Other
Solid-solution alloys												
Iron-nickel-base												
Alloy N-155 (Multimet)	R30155	21.0	20.0	20.0	3.00	2.5	1.0	—	—	32.2	0.15	0.15 N, 0.2 La, 0.02 Zr
Haynes 556	R30556	22.0	21.0	20.0	3.0	2.5	0.1	—	0.3	29.0	0.10	0.50 Ta, 0.02 La, 0.002 Zr
19-9 DL	S63198	19.0	9.0	—	1.25	1.25	0.4	0.3	—	66.8	0.30	1.10 Mn, 0.60 Si
Incoloy 800	N08800	21.0	32.5	—	—	—	—	0.38	0.38	45.7	0.05	—
Incoloy 800H	N08810	21.0	33.0	—	—	—	—	—	—	45.8	0.08	—
Incoloy 800HT	N08811	21.0	32.5	—	—	—	—	0.4	0.4	46.0	0.08	0.8 Mn, 0.5 Si, 0.4 Cu
Incoloy 801	N08801	20.5	32.0	—	—	—	—	1.13	—	46.3	0.05	—
Incoloy 802	—	21.0	32.5	—	—	—	—	0.75	0.58	44.8	0.35	—
Nickel-base												
Haynes 214	—	16.0	76.5	—	—	—	—	—	4.5	3.0	0.03	—
Haynes 230	N06230	22.0	55.0	5.0 max	2.0	14.0	—	—	0.35	3.0 max	0.10	0.015 max B, 0.02 La
Inconel 660	N06600	15.5	76.0	—	—	—	—	—	—	8.0	0.08	0.25 Cu
Inconel 601	N06601	23.0	60.5	—	—	—	—	—	1.35	14.1	0.05	0.5 Cu
Inconel 617	N06617	22.0	55.0	12.5	9.0	—	—	—	1.0	—	0.07	—
Inconel 625	N06625	21.5	61.0	—	9.0	—	3.6	0.2	0.2	2.5	0.05	—
RA 333	N06333	25.0	45.0	3.0	3.0	3.0	—	—	—	18.0	0.05	—
Hastelloy B	N10001	1.0 max	63.0	2.5 max	28.0	—	—	—	—	5.0	0.05 max	0.03 V
Hastelloy N	N10003	7.0	72.0	—	16.0	—	—	0.5 max	—	5.0 max	0.06	—
Hastelloy S	N06635	15.5	67.0	—	15.5	—	—	—	0.2	1.0	0.02 max	0.02 La
Hastelloy W	N10004	5.0	61.0	2.5 max	24.5	—	—	—	—	5.5	0.12 max	0.6 V
Hastelloy X	N06002	22.0	49.0	1.5 max	9.0	0.6	—	—	2.0	15.8	0.15	—
Hastelloy C-276	N10276	15.5	59.0	—	16.0	3.7	—	—	—	5.0	0.02 max	—
Haynes HR-120	N08120	25.0	37.0	3.0	2.5	2.5	0.7	—	0.1	33.0	0.05	0.7 Mn, 0.6 Si, 0.2 N, 0.004 B
Haynes HR-160	N12160	28.0	37.0	29.0	—	—	—	—	—	2.0	0.05	2.75 Si, 0.5 Mn
Nimonic 75	N06075	19.5	75.0	—	—	—	—	0.4	0.15	2.5	0.12	0.25 max Cu
Nimonic 86	—	25.0	65.0	—	10.0	—	—	—	—	—	0.05	0.03 Ce, 0.015 Mg
Cobalt-base												
Haynes 25 (L605)	R30605	20.0	10.0	50.0	—	15.0	—	—	—	3.0	0.10	1.5 Mn
Haynes 188	R30188	22.0	22.0	37.0	—	14.5	—	—	—	3.0 max	0.10	0.90 La
Alloy S-816	R30816	20.0	20.0	42.0	4.0	4.0	4.0	—	—	4.0	0.38	—
MP35-N	R30035	20.0	35.0	35.0	10.0	—	—	—	—	—	—	—

MP159	R30159	19.0	25.0	36.0	7.0	—	0.6	3.0	0.2	9.0	—	—
Stellite B	N07718	30.0	1.0	61.5	—	4.5	—	—	—	1.0	1.0	—
UMCo-50	—	28.0	—	49.0	—	—	—	—	—	21.0	0.12	—
Precipitation-hardening alloys												
Iron-nickel-base												
A-286	S66286	15.0	26.0	—	1.25	—	—	2.0	0.2	55.2	0.04	0.005 B, 0.3 V
Discaloy	S66220	14.0	26.0	—	3.0	—	—	1.7	0.25	55.0	0.06	—
Incoloy 903	N19903	0.1 max	38.0	15.0	0.1	—	3.0	1.4	0.7	41.0	0.04	—
Pyromet CTX-1	—	0.1 max	37.7	16.0	0.1	—	3.0	1.7	1.0	39.0	0.03	—
Incoloy 907	N19907	—	38.4	13.0	—	—	4.7	1.5	0.03	42.0	0.01	0.15 Si
Incoloy 909	N19909	—	38.0	13.0	—	—	4.7	1.5	0.03	42.0	0.01	0.4 Si
Incoloy 925	N09925	20.5	44.0	—	2.8	—	—	2.1	0.2	29	0.01	1.8 Cu
V-57	—	14.8	27.0	—	1.25	—	—	3.0	0.25	48.6	0.08 max	0.01 B, 0.5 max V
W-545	S66545	13.5	26.0	—	1.5	—	—	2.85	0.2	55.8	0.08 max	0.05 B
Nickel-base												
Astroloy	N13017	15.0	56.5	15.0	5.25	—	—	3.5	4.4	<0.3	0.06	0.03 B, 0.06 Zr
Custom Age 625 PLUS	N07716	21.0	61.0	—	8.0	—	3.4	1.3	0.2	5.0	0.01	—
Haynes 242	—	8.0	62.5	2.5 max	25.0	—	—	—	0.5 max	2.0 max	0.10 max	0.006 max B
Haynes 263	N07263	20.0	52.0	—	6.0	—	—	2.4	0.6	0.7	0.06	0.6 Mn, 0.4 Si, 0.2 Cu
Haynes R-41	N07041	19.0	52.0	11.0	10.0	—	—	3.1	1.5	5.0	0.09	0.5 Si, 0.1 Mn, 0.006 B
Inconel 100	N13100	10.0	60.0	15.0	3.0	—	—	4.7	5.5	<0.6	0.15	1.0 V, 0.06 Zr, 0.015 B
Inconel 102	N06102	15.0	67.0	—	2.9	3.0	2.9	0.5	0.5	7.0	0.06	0.005 B, 0.02 Mg, 0.03 Zr
Incoloy 901	N09901	12.5	42.5	—	6.0	—	—	2.7	—	36.2	0.10 max	—
Inconel 702	N07702	15.5	79.5	—	—	—	—	0.6	3.2	1.0	0.05	0.5 Mn, 0.2 Cu, 0.4 Si
Inconel 706	N09706	16.0	41.5	—	—	—	—	1.75	0.2	37.5	0.03	2.9 (Nb + Ta), 0.15 max Cu
Inconel 718	N07718	19.0	52.5	—	3.0	—	5.1	0.9	0.5	18.5	0.08 max	0.15 max Cu
Inconel 721	N07721	16.0	71.0	—	—	—	—	3.0	—	6.5	0.04	2.2 Mn, 0.1 Cu
Inconel 722	N07722	15.5	75.0	—	—	—	—	2.4	0.7	7.0	0.04	0.5 Mn, 0.2 Cu, 0.4 Si
Inconel 725	N07725	21.0	57.0	—	8.0	—	3.5	1.5	0.35 max	9.0	0.03 max	—
Inconel 751	N07751	15.5	72.5	—	—	—	1.0	2.3	1.2	7.0	0.05	0.25 max Cu
Inconel X-750	N07750	15.5	73.0	—	—	—	1.0	2.5	0.7	7.0	0.04	0.25 max Cu
M-252	N07252	19.0	56.5	10.0	10.0	—	—	2.6	1.0	<0.75	0.15	0.005 B
Nimonic 80A	N07080	19.5	73.0	1.0	—	—	—	2.25	1.4	1.5	0.05	0.10 max Cu
Nimonic 90	N07090	19.5	55.5	18.0	—	—	—	2.4	1.4	1.5	0.06	—
Nimonic 95	—	19.5	53.5	18.0	—	—	—	2.9	2.0	5.0 max	0.15 max	+B, +Zr
Nimonic 100	—	11.0	56.0	20.0	5.0	—	—	1.5	5.0	2.0 max	0.30 max	+B, +Zr
Nimonic 105	—	15.0	54.0	20.0	5.0	—	—	1.2	4.7	—	0.08	0.005 B
Nimonic 115	—	15.0	55.0	15.0	4.0	—	—	4.0	5.0	1.0	0.20	0.04 Zr

Table 3 (*Continued*)

Alloy	UNS Number	Composition (%)										
		Cr	Ni	Co	Mo	W	Nb	Ti	Al	Fe	C	Other
C-263	N07263	20.0	51.0	20.0	5.9	—	—	2.1	0.45	0.7 max	0.06	—
Pyromet 860	—	13.0	44.0	4.0	6.0	—	—	3.0	1.0	28.9	0.05	0.01 B
Pyromet 31	N07031	22.7	55.5	—	2.0	—	1.1	2.5	1.5	14.5	0.04	0.005 B
Refractaloy 26	—	18.0	38.0	20.0	3.2	—	—	2.6	0.2	16.0	0.03	0.015 B
René 41	N07041	19.0	55.0	11.0	10.0	—	—	3.1	1.5	<0.3	0.09	0.01 B
René 95	—	14.0	61.0	8.0	3.5	3.5	3.5	2.5	3.5	<0.3	0.16	0.01 B, 0.05 Zr
René 100	—	9.5	61.0	15.0	3.0	—	—	4.2	5.5	1.0 max	0.16	0.015 B, 0.06 Zr, 1.0 V
Udimet 500	N07500	19.0	48.0	19.0	4.0	—	—	3.0	3.0	4.0 max	0.08	0.005 B
Udimet 520	—	19.0	57.0	12.0	6.0	1.0	—	3.0	2.0	—	0.08	0.005 B
Udimet 630	—	17.0	50.0	—	3.0	3.0	6.5	1.0	0.7	18.0	0.04	0.004 B
Udimet 700	—	15.0	53.0	18.5	5.0	—	—	3.4	4.3	<1.0	0.07	0.03 B
Udimet 710	—	18.0	55.0	14.8	3.0	1.5	—	5.0	2.5	—	0.07	0.01 B
Unitemp AF2-1DA	N07012	12.0	59.0	10.0	3.0	6.0	—	3.0	4.6	<0.5	0.35	1.5 Ta, 0.015 B, 0.1 Zr
Waspaloy	N07001	19.5	57.0	13.5	4.3	—	—	3.0	1.4	2.0 max	0.07	0.006 B, 0.09 Zr

Source: *Metals Handbook Desk Edition,* ASM International, Materials Park, OH 44073-0002, 1999, p. 397.

Table 4 Nominal Compositions of Cast Superalloys

Alloy Designation	Nominal Composition (%)											
	C	Ni	Cr	Co	Mo	Fe	Al	B	Ti	Ta	W	Zr	Other
Nickel Base													
B-1900	0.1	64	8	10	6	—	6	0.015	1	4[a]	—	0.10	—
CMSX-2	—	66.2	8	4.6	0.6	—	56	—	1	6	8	6	—
Hastelloy X	0.1	50	21	1	9	18	—	—	—	—	1	—	—
Inconel 100	0.18	60.5	10	15	3	—	5.5	0.01	5	—	—	0.06	1 V
Inconel 713C	0.12	74	12.5	—	4.2	—	6	0.012	0.8	1.75	—	0.1	0.9 Nb
Inconel 713LC	0.05	75	12	—	4.5	—	6	0.01	0.6	4	—	0.1	—
Inconel 738	0.17	61.5	16	8.5	1.75	—	3.4	0.01	3.4	—	2.6	0.1	2 Nb
Inconel 792	0.2	60	13	9	2.0	—	3.2	0.02	4.2	—	4	0.1	2 Nb
Inconel 718	0.04	53	19	—	3	18	0.5	—	0.9	—	—	—	0.1 Cu, 5 Nb
X-750	0.04	73	15	—	—	7	0.7	—	2.5	—	—	—	0.25 Cu, 0.9 Nb
M-252	0.15	56	20	10	10	—	1	0.005	2.6	—	—	—	—
MAR-M 200	0.15	59	9	10	—	1	5	0.015	2	—	12.5	0.05	1 Nb[b]
MAR-M 246	0.15	60	9	10	2.5	—	5.5	0.015	1.5	1.5	10	0.05	—
MAR-M 247	0.15	59	8.25	10	0.7	0.5	5.5	0.015	1	3	10	0.05	1.5 Hf
PWA 1480	—	bal	10	5.0	—	—	5.0	—	1.5	12	4.0	—	—
René 41	0.09	55	19	11.0	10.0	—	1.5	0.01	3.1	—	—	—	—
René 77	0.07	58	15	15	4.2	—	4.3	0.015	3.3	—	—	0.04	—
René 80	0.17	60	14	9.5	4	—	3	0.015	5	—	4	0.03	—
René 80 Hf	0.08	60	14	9.5	4	—	3	0.015	4.8	—	4	0.02	0.75 Hf
René 100	0.18	61	9.5	15	3	—	5.5	0.015	4.2	—	—	0.06	1 V
René N4	0.06	62	9.8	7.5	1.5	—	4.2	0.004	3.5	4.8	6	—	0.5 Nb, 0.15 Hf
Udimet 500	0.1	53	18	17	4	2	3	—	3	—	—	—	—
Udimet 700	0.1	53.5	15	18.5	5.25	—	4.25	0.03	3.5	—	—	—	—
Udimet 710	0.13	55	18	15	3	—	2.5	—	5	—	1.5	0.08	—
Waspaloy	0.07	57.5	19.5	13.5	4.2	1	1.2	0.005	3	—	—	0.09	—
WAX-20 (DS)	0.20	72	—	—	—	—	6.5	—	—	—	20	1.5	—
Cobalt Base													
AiResist 13	0.45	—	21	62	—	—	3.4	—	—	2	11	—	0.1 Y
AiResist 213	0.20	0.5	20	64	—	0.5	3.5	—	—	6.5	4.5	0.1	0.1 Y
AiResist 215	0.35	0.5	19	63	—	0.5	4.3	—	—	7.5	4.5	0.1	0.1 Y
FSX-414	0.25	10	29	52.5	—	1	—	0.010	—	—	7.5	—	—
Haynes 21	0.25	3	27	64	—	1	—	—	—	—	—	—	5 Mo
Haynes 25; L-605	0.1	10	20	54	—	1	—	—	—	—	15	—	—
J-1650	0.20	27	19	36	—	—	—	0.02	3.8	2	12	—	—
MAR-M 302	0.85	—	21.5	58	—	0.5	—	0.005	—	9	10	0.2	—
MAR-M 322	1.0	—	21.5	60.5	—	0.5	—	—	0.75	4.5	9	2	—
MAR-M 509	0.6	10	23.5	54.5	—	—	—	—	0.2	3.5	7	0.5	—
MAR-M 918	0.05	20	20	52	—	—	—	—	—	7.5	—	0.1	—
NASA Co-W-Re	0.40	—	3	67.5	—	—	—	—	1	—	25	1	2 Re
S-816	0.4	20	20	42	—	4	—	—	—	—	4	—	4 Mo, 4 Nb, 1.2 Mn, 0.4 Si
V-36	0.27	20	25	42	—	3	—	—	—	—	2	—	4 Mo, 2 Nb, 1 Mn, 0.4 Si
WI-52	0.45	—	21	63.5	—	2	—	—	—	—	11	—	2 Nb + Ta
X-40 (Stellite alloy 31)	0.50	10	22	57.5	—	1.5	—	—	—	—	7.5	—	0.5 Mn, 0.5 Si

[a] B-1900 + Hf also contains 1.5% Hf.

[b] MAR-M 200 + Hf also contains 1.5% Hf.

Source: *Metals Handbook Desk Edition,* ASM International, Materials Park, OH 44073-0002, 1999, p. 399.

Table 5 Physical Properties of Cast Nickel-Base and Cobalt-Base Alloys

				Specific Heat						Thermal Conductivity								
		Melting Range		At 21°C (70°F)		At 538°C (1000°F)		At 1093°C (2000°F)		At 93°C (200°F)		At 538°C (1000°F)		At 1093°C (2000°F)		Mean Coefficient of Thermal Expansion (10^{-6}/K)[a]		
Alloy	Density (g/cm³)	°C	°F	J/ kg · K	Btu/ lb · °F	J/ kg · K	Btu/ lb · °F	J/ kg · K	Btu/ lb · °F	W/ m · K	Btu · in/ h · ft² · °F	W/ m · K	Btu · in/ h · ft² · °F	W/ m · K	Btu · in/ h · ft² · °F	At 93°C (200°F)	At 538°C (1000°F)	At 1093°C (2000°F)
									Nickel Base									
IN-713 C	7.91	1260–1290	2300–2350	420	0.10	565	0.135	710	0.17	10.9	76	17.0	118	26.4	183	10.6	13.5	17.1
IN-713 LC	8.00	1290–1320	2350–2410	440	0.105	565	0.135	710	0.17	10.7	74	16.7	116	25.3	176	10.1	15.8	18.9
B-1900	8.22	1275–1300	2325–2375	—	—	—	—	—	—	(10.2)	(71)	16.3	113	—	—	11.7	13.3	16.2
Cast alloy 625	8.44	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
Cast alloy 718	8.22	1205–1345	2200–2450	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
IN-100	7.75	1265–1335	2305–2435	—	—	480	0.115	605	0.145	—	—	17.3	120	—	—	13.0	13.9	18.1
IN-162	8.08	1275–1305	2330–2380	—	—	—	—	—	—	—	—	—	—	—	12.2	14.1	—	
IN-731	7.75	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
IN-738	8.11	1230–1315	2250–2400	420	0.10	565	0.135	710	0.17	—	—	17.7	123	27.2	189	11.6	14.0	—
IN-792	8.25	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
M-22	8.63	—	—	—	—	—	—	—	—	—	—	—	—	—	—	12.4	13.3	—
MAR-M 200	8.53	1315–1370	2400–2500	400	0.095	420	0.10	565	0.135	13.0	90	15.2	110	29.7	206	—	13.1	17.0
MAR-M 246	8.44	1315–1345	2400–2450	—	—	—	—	—	—	—	—	18.9	131	30.0	208	11.3	14.8	18.6
MAR-M 247	8.53	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
MAR-M 421	8.08	—	—	—	—	—	—	—	—	—	—	19.1	137	32.0	229	—	14.9	19.8
MAR-M 432	8.16	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	14.9	19.3
MC-102	8.84	—	—	—	—	—	—	—	—	—	—	—	—	—	—	12.8	14.9	—
Nimocast 75	8.44	1410[b]	2570[b]	—	—	—	—	—	—	—	—	—	—	—	—	12.8	14.9	—
Nimocast 80	8.17	1310–1380	2390–2515	—	—	—	—	—	—	—	—	—	—	—	—	12.8	14.9	—
Nimocast 90	8.18	1310–1380	2390–2515	—	—	—	—	—	—	—	—	—	—	—	—	12.3	14.8	—
Nimocast 242	8.40	1225–1340	2235–2445	—	—	—	—	—	—	—	—	—	—	—	—	12.5	14.4	—
Nimocast 263	8.36	1300–1355	2370–2470	—	—	—	—	—	—	—	—	—	—	—	—	11.0	13.6	—
René 77	7.91	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
René 80	8.16	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
Udimet 500	8.02	1300–1395	2375–2540	—	—	—	—	—	—	—	—	—	—	—	—	13.3	—	—
Udimet 710	8.08	—	—	—	—	—	—	—	—	12.1	84	18.1	126	—	—	—	—	—

Cobalt Base																		
FSX-414	8.3	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
Haynes 1002	8.75	1305–1420	2380–2590	420	0.10	530	0.126	645	0.154	11.0	76	21.8	151	32.1	222	12.2	14.4	—
MAR-M 302	9.21	1315–1370	2400–2500	—	—	—	—	—	—	18.7	130	22.2	154	—	—	—	13.7	16.6
MAR-M 322	8.91	1315–1360	2400–2475	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
MAR-M 509	8.85	—	—	—	—	—	—	—	—	—	—	27.9	194	44.6	310	9.8	15.9	18.2
WI-52	8.88	1300–1355	2425–2475	420	0.10	—	—	—	—	24.8	172	27.4	190	40.3	280	—	14.4	17.5
X-40	8.60	—	—	—	—	—	—	—	—	11.8	82	21.6	150	—	—	—	15.1	—

[a] From room temperature to indicated temperature.

[b] Liquidus temperature.

Source: From *Advanced Materials and Processes,* ASM International, Materials Park, OH 44073-0002, 2000, p. 91.

Table 6 Effect of Temperature on the Mechanical Properties of Wrought Nickel-, Iron-, and Cobalt-Base Superalloys

		Ultimate Tensile Strength at:						Yield Strength at 0.2% offset:						Tensile eleongation, % at:		
		21°C (70°F)		540°C (1000°F)		760°C (1400°F)		21°C (70°F)		540°C (1000°F)		760°C (1400°F)				
Alloy	Form	MPa	ksi	MPa	ksi	MPa	ksi	MPa	ksi	MPa	ksi	MPa	ksi	21°C (70°F)	540°C (1000°F)	760°C (1400°F)
Nickel Base																
Astroloy	Bar	1415	205	1240	180	1160	168	1050	152	965	140	910	132	16	16	21
Cabot 214	—	915	133	715	104	560	84	560	81	510	74	495	72	38	19	9
D-979	Bar	1401	204	1295	188	720	104	1005	146	925	134	655	95	15	15	17
Hastelloy C-22	Sheet	800	116	625	91	525	76	405	59	275	40	240	35	57	61	63
Hastelloy G-30	Sheet	690	100	490	71	—	—	315	46	170	25	—	—	64	75	—
Hastelloy S	Bar	845	130	775	112	575	84	455	65	340	49	310	45	49	50	70
Hastelloy X	Sheet	785	114	650	94	435	63	360	52	290	42	260	38	43	45	37
Haynes 230	*a*	870	126	720	105	575	84	390	57	275	40	285	41	48	56	46
Inconel 587	Bar	1180	171	1035	150	830	120	705	102	620	90	605	88	28	22	20
Inconel 597	Bar	1220	177	1140	165	930	135	760	110	720	104	665	96	15	15	16
Inconel 600	Bar	660	96	560	81	260	38	285	41	220	32	180	26	45	41	70
Inconel 601	Sheet	740	107	725	105	290	42	455	66	350	51	220	32	40	34	78
Inconel 617	Bar	740	107	580	84	440	64	295	43	200	29	180	26	70	68	84
Inconel 617	Sheet	770	112	590	86	470	68	345	50	230	33	230	33	55	62	59
Inconel 625	Bar	965	140	910	132	550	80	490	71	415	60	415	60	50	50	45
Inconel 706	Bar	1310	190	1145	166	725	105	1005	146	910	132	660	96	20	19	32
Inconel 718	Bar	1435	208	1275	185	950	138	1185	172	1065	154	740	107	21	18	25
Inconel 718 Direct Age	Bar	1530	222	1350	196	—	—	1365	198	1180	171	—	—	16	15	—
Inconel 718 Super	Bar	1350	196	1200	174	—	—	1105	160	1020	148	—	—	16	18	—
Inconel X750	Bar	1200	174	1050	152	—	—	815	118	725	105	—	—	27	26	—
M-252	Bar	1240	180	1230	178	945	137	840	122	765	111	720	104	16	15	10
Nimonic 75	Bar	745	108	675	98	310	45	285	41	200	29	160	23	40	40	67
Nimonic 80A	Bar	1000	145	875	127	600	87	620	90	530	77	505	73	39	37	17
Nimonic 90	Bar	1235	179	1075	156	655	95	810	117	725	105	540	78	33	28	12
Nimonic 105	Bar	1180	171	1130	164	930	135	830	120	775	112	740	107	16	22	25
Nimonic 115	Bar	1240	180	1090	158	1085	157	865	125	795	115	800	116	27	18	24
Nimonic 263	Sheet	970	141	800	116	650	94	580	84	485	70	460	67	39	42	21
Nimonic 942	Bar	1405	204	1300	189	900	131	1060	154	970	141	860	125	37	26	42
Nimonic PE, 11	Bar	1080	157	1000	145	760	110	720	105	690	100	560	81	30	30	18
Nimonic PE 16	Bar	885	128	740	107	510	74	530	77	485	70	370	54	37	26	42
Nimonic PK 33	Sheet	1180	171	1000	145	885	128	780	113	725	105	670	97	30	30	18
Pyromet 860	Bar	1295	188	1255	182	910	132	835	121	840	122	835	121	22	15	18
René 41	Bar	1420	206	1400	203	1105	160	1060	154	1020	147	940	136	14	14	11
René 95	Bar	1620	235	1550	224	1170	170	1310	190	1255	182	1100	160	15	12	15
Udimet 400	Bar	1310	190	1185	172	—	—	930	135	830	120	—	—	30	26	—
Udimet 500	Bar	1310	190	1240	180	1040	151	840	122	795	115	730	106	32	28	39
Udimet 520	Bar	1310	190	1240	180	725	105	860	125	825	120	725	105	21	20	15
Udimet 630	Bar	1520	220	1380	200	965	140	1310	190	1170	170	860	125	15	15	5

Udimet 700	Bar	1410	204	1275	185	1035	150	965	140	895	130	825	120	17	16	20
Udimet 710	Bar	1185	172	1150	167	1020	148	910	132	850	123	815	118	7	10	25
Udimet 720	Bar	1570	228	—	—	1455	211	1195	173	—	—	1050	152	13	—	9
Unitemp AF2-1DA6	Bar	1560	226	1480	215	1290	187	1015	147	1040	151	995	144	20	19	16
Waspaloy	Bar	1275	185	1170	170	650	94	795	115	725	105	675	98	25	23	28
Iron Base																
A-286	Bar	1005	146	905	131	440	64	725	105	605	88	430	62	25	19	19
Alloy 901	Bar	1205	175	1030	149	725	105	895	130	780	113	635	92	14	14	19
Discaloy	Bar	1000	145	865	125	485	70	730	106	650	94	430	62	19	16	—
Haynes 556	Sheet	815	118	645	93	470	69	410	60	240	35	220	32	48	54	49
Incoloy 800	Bar	595	86	510	74	235	34	250	36	180	26	150	22	44	38	83
Incoloy 801	Bar	785	114	660	96	325	47	385	56	310	45	290	42	30	28	55
Incoloy 802	Bar	690	100	600	87	400	58	290	42	195	28	200	29	44	39	15
Incoloy 807	Bar	655	95	470	68	350	51	380	55	255	37	225	32.5	48	40	34
Incoloy 825[b]	—	690	100	~590	~86	~275	~40	310	45	~234	~34	180	~26	45	~44	~86
Incoloy 903	Bar	1310	190	—	—	—	—	1105	160	—	—	—	—	14	—	—
Incoloy 907[c]	—	~1365	~198	~1205	~175	~655	~95	~1110	~161	~960	~139	~565	~82	~12	~11	~20
Incoloy 909	Bar	1310	190	1160	168	615	89	1020	148	945	137	540	78	16	14	34
N-155	Bar	815	118	650	94	428	62	400	58	340	49	250	36	40	33	32
V-57	Bar	1170	170	1000	145	620	90	830	120	760	110	485	70	26	19	34
19-9 DL	—	815	118	615	89	—	—	570	83	395	57	—	—	43	30	—
16-25-6	—	980	142	—	—	415	60	770	112	—	—	345	50	23	—	11
Cobalt Base																
AirResist 213	—	1120	162	—	—	485	70	625	91	—	—	385	56	14	—	47
Elgiloy	—	690[b] 2480[d]	100[b] 360[d]	—	—	—	—	480[b] 2000[d]	70–290	—	—	—	—	34	—	—
Haynes 188	Sheet	960	139	740	107	635	92	485	70	305	44	290	42	56	70	43
L-605	Sheet	1005	146	800	116	455	66	460	67	250	36	260	38	64	59	12
MAR-M 918	Sheet	895	130	—	—	—	—	895	130	—	—	—	—	48	—	—
MP35N	Bar	2025	294	—	—	—	—	1620	235	—	—	—	—	10	—	—
MP159	Bar	1895	275	1565	227	—	—	1825	265	1495	217	—	—	8	8	—
Stellite 6B	Sheet	1010	146	—	—	—	—	635	92	—	—	—	—	11	—	—
Haynes 150	—	925	134	—	—	—	—	317	46	—	—	—	—	8	—	—

[a] Cold-rolled and solution-annealed sheet, 1.2–1.6 mm (0.048–0.063 in.) thick.

[b] Annealed.

[c] Precipitation hardened.

[d] Work strengthened and aged.

Source: *Metals Handbook Desk Edition,* ASM International, Materials Park, OH 44073-0002, 1999, p. 409.

Table 7 Effect of Temperature on the Mechanical Properties of Cast Nickel-Base and Cobalt-Base Alloys

	Ultimate Tensile Strength at:						0.2% Yield Strength at:						Tensile Elongation, % at:		
	21°C (70°F)		538°C (1000°F)		1093°C (2000°F)		21°C (70°F)		538°C (1000°F)		1093°C (2000°F)				
Alloy	MPa	ksi	MPa	ksi	MPa	ksi	MPa	ksi	MPa	ksi	MPa	ksi	21°C (70°F)	538°C (1000°F)	1093°C (2000°F)
Nickel Base															
IN-713 C	850	123	860	125	—	—	740	107	705	102	—	—	8	10	—
IN-713 LC	895	130	895	130	—	—	750	109	760	110	—	—	15	11	—
B-1900	970	141	1005	146	270	38	825	120	870	126	195	28	8	7	11
IN-625	710	103	510	74	—	—	350	51	235	34	—	—	48	50	—
IN-718	1090	158	—	—	—	—	915	133	—	—	—	—	11	—	—
IN-100	1018	147	1090	150	(380)	(55)	850	123	885	128	(240)	(35)	9	9	—
IN-162	1005	146	1020	148	—	—	815	118	795	115	—	—	7	6.5	—
IN-731	835	121	—	—	275	40	725	105	—	—	170	25	6.5	—	—
IN-738	1095	159	—	—	—	—	950	138	—	—	—	—	—	—	—
IN-792	1170	170	—	—	—	—	1060	154	—	—	—	—	4	—	—
M-22	730	106	780	113	—	—	685	99	730	106	—	—	5.5	4.5	—
MAR-M 200	930	135	945	137	325	47	840	122	880	123	—	—	7	5	—
MAR-M 246	965	140	1000	145	345	50	860	125	860	125	—	—	5	5	—
MAR-M 247	965	140	1035	150	—	—	815	118	825	120	—	—	7	—	—
MAR-M 421	1085	157	995	147	—	—	930	135	815	118	—	—	4.5	3	—
MAR-M 432	1240	180	1105	160	—	—	1070	155	910	132	—	—	6	—	—
MC-102	675	98	655	95	—	—	605	88	540	78	—	—	5	9	—
Nimocast 75	500	72	—	—	—	—	179	26	—	—	—	—	39	—	—
Nimocast 80	730	106	—	—	—	—	520	75	—	—	—	—	15	—	—
Nimocast 90	700	102	595	86	—	—	520	75	420	61	—	—	14	15	—
Nimocast 242	460	67	—	—	—	—	300	44	—	—	—	—	8	—	—
Nimocast 263	730	106	—	—	—	—	510	74	—	—	—	—	18	—	—
René 77	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
René 80	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
Udimet 500	930	135	895	130	—	—	815	118	725	105	—	—	13	13	—
Udimet 710	1075	156	—	—	240	35	895	130	—	—	170	25	8	—	—
CMSX-2[a]	1185	172	1295^b	188^b	—	—	1135	165	1245^b	181^b	—	—	10	17^b	—
GMR-235	710	103	—	—	—	—	640	93	—	—	—	—	3	—	18^c
IN-939	1050	152	915^b	133^b	325^c	47^c	80	116	635^b	92^b	205^c	30^c	5	7^b	25^c
MM 002[d]	1035	150	1035^b	150^b	550^c	85^c	825	120	860^b	125^b	345^c	50^c	7	5^b	12^c

IN-713 Hf[e]	1000	145	895[b]	130[b]	380[c]	55[c]	760	110	620[b]	90[b]	240[c]	35[c]	11	6[b]	20[c]
René 125 Hf[f]	1070	155	1070[b]	155[b]	550[c]	80[c]	825	120	860[b]	125[b]	345[c]	50[c]	5	5[b]	12[c]
MAR-M 246 Hf[g]	1105	160	1070[b]	155[b]	565[c]	82[c]	860	125	860[b]	125[b]	345[c]	50[c]	6	7[b]	14[c]
MAR-M 200 Hf[h]	1035	150	1035[b]	150[b]	540[c]	78[c]	825	120	860[b]	125[b]	345[c]	50[c]	5	5[b]	10[c]
PWA-1480[a]	—	—	1130[b]	164[b]	685[c]	99[c]	895	130	905[b]	131[b]	495[c]	72[c]	4	8[b]	20[c]
SEL	1020	148	875[b]	127[b]	—	—	905	131	795[b]	115[b]	—	—	6	7[b]	—
UDM 56	945	137	945[b]	137[b]	—	—	850	123	725[b]	105[b]	—	—	3	5[b]	—
SEL-15	1060	154	1090[b]	158[b]	—	—	895	130	815[b]	118[b]	—	—	9	5[b]	—
Cobalt Base															
AiResist 13[i]	600	87	420[b]	61[b]	—	—	530	77	330[b]	48[b]	—	—	1.5	4.5[b]	—
AiResist 215[i]	690	100	570[j]	83[j]	—	—	485	70	315[j]	46[j]	—	—	4	12[j]	—
FSX-414	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
Haynes 1002	770	112	560	81	115	17	470	68	345	50	95	14	6	8	28
MAR-M 302	930	135	795	115	150	22	690	100	505	73	150	22	2	—	21
MAR-M 322[i]	830	120	595[b]	86[b]	—	—	630	91	345[b]	50[b]	—	—	4	6.5[b]	—
MAR-M 509	785	114	570	83	—	—	570	83	400	58	—	—	4	6	—
WI-52	750	109	745	108	160	23	585	85	440	64	105	15	5	7	35
X-40	745	108	550	80	—	—	525	76	275	40	—	—	9	17	—

[a] Single crystal [001].

[b] At 760°C (1400°F).

[c] At 980°C (1800°F).

[d] RR-7080.

[e] MM 004.

[f] MM 005.

[g] MM 006.

[h] MM 009.

[i] Data from Vol. 3, 9th ed., *Metals Handbook,* 1980.

[j] At 650°C (1200°F).

Source: *Metals Handbook Desk Edition,* ASM International, Materials Park, OH 44073-0002, 1999, p. 409.

Table 8 1000-h Rupture Strengths of Wrought Nickel-, Cobalt, and Iron-Base Superalloys

		Rupture Strength at:							
		650°C (1200°F)		760°C (1400°F)		870°C (1600°F)		980°C (1800°F)	
Alloy	Form	MPa	ksi	MPa	ksi	MPa	ksi	MPa	ksi
Nickel Base									
Astroloy	Bar	770	112	425	62	170	25	55	8
Cabot 214	—	—	—	—	—	30	4	15	2
D-979	Bar	515	75	250	36	70	10	—	—
Hastelloy S	Bar	—	—	90	13	25	4	—	—
Hastelloy X	Sheet	215	31	105	15	40	6	15	2
Haynes 230	—	—	—	125	18	55	8	15	2
Inconel 587	Bar	—	—	285	41	—	—	—	—
Inconel 597	Bar	—	—	340	49	—	—	—	—
Inconel 600	Bar	—	—	—	—	30	4	15	2
Inconel 601	Sheet	195	28	60	9	30	4	15	2
Inconel 617	Bar	360	52	165	24	60	9	30	4
Inconel 617	Sheet	—	—	160	23	60	9	30	4
Inconel 625	Bar	370	54	160	23	50	7	20	3
Inconel 706	Bar	580	84	—	—	—	—	—	—
Inconel 718	Bar	595	86	195	28	—	—	—	—
Inconel 718 Direct Age	Bar	405	59	—	—	—	—	—	—
Inconel 718 Super	Bar	600	87	—	—	—	—	—	—
Inconel X750	Bar	470	68	—	—	50	7	—	—
M-252	Bar	565	82	270	39	95	14	—	—
Nimonic 75	Bar	170	25	50	7	5	1	—	—
Nimonic 80A	Bar	420	61	160	23	—	—	—	—
Nimonic 90	Bar	455	66	205	30	60	9	—	—
Nimonic 105	Bar	—	—	330	48	130	19	30	4
Nimonic 115	Bar	—	—	420	61	185	27	70	10
Nimonic 942	Bar	520	75	270	39	—	—	—	—
Nimonic PE 11	Bar	335	49	145	21	—	—	—	—
Nimonic PE 16	Bar	345	50	150	22	—	—	—	—
Nimonic PK 33	Sheet	655	95	310	45	90	13	—	—
Pyromet 860	Bar	545	79	250	36	—	—	—	—
René 41	Bar	705	102	345	50	115	17	—	—
René 95	Bar	860	125	—	—	—	—	—	—

Udimet 400	Bar	600	87	305	44	110	16	—	—
Udimet 500	Bar	760	110	325	47	125	18	—	—
Udimet 520	Bar	585	85	345	50	150	22	—	—
Udimet 700	Bar	705	102	425	62	200	29	55	8
Udimet 710	Bar	870	126	460	67	200	29	70	10
Udimet 720	Bar	670	97	—	—	—	—	—	—
Unitemp AF2-1DA6	Bar	885	128	360	52	—	—	—	—
Waspaloy	Bar	615	89	290	42	110	16	—	—
Iron Base									
A-286	Bar	315	46	105	15	—	—	—	—
Alloy 901	Sheet	525	76	205	30	—	—	—	—
Discaloy	Bar	275	40	60	9	—	—	—	—
Haynes 556	Sheet	275	40	125	18	55	8	20	3
Incoloy 800	Bar	165	24	66	9.5	30	4.4	13	1.9
Incoloy 801	Bar	—	—	—	—	—	—	—	—
Incoloy 802	Bar	170	25	110	16	69	10	24	3.5
Incoloy 807	Bar	—	—	105	15	43	6.2	19	2.7
Incoloy 903	Bar	510	74	—	—	—	—	—	—
Incoloy 909	Bar	345	50	—	—	—	—	—	—
N-155	Bar	295	43	140	20	70	10	20	3
V-57	Bar	485	70	—	—	—	—	—	—
Cobalt Base									
Haynes 188	Sheet	—	—	165	24	70	10	30	4
L-605	Sheet	270	39	165	24	75	11	30	4
MAR-M 918	Sheet	—	—	60	9	20	3	5	1
Haynes 150	—	—	—	40[a]	5.8	—	—	—	—

[a] At 815°C (1500°F).

Source: *Metals Handbook Desk Edition,* ASM International, Materials Park, OH 44073-0002, 1999, p. 410.

Table 9 Stress-Rupture Strengths for Selected Cast Nickel-Base Superalloys

	Rupture Stress at:					
	815°C (1500°F)		870°C (1600°F)		980°C (1800°F)	
Alloy	100 h MPa (ksi)	1000 h MPa (ksi)	100 h MPa (ksi)	1000 h MPa (ksi)	100 h MPa (ksi)	1000 h MPa (ksi)
Nickel Base						
IN-713 LC	425 (62)	325 (47)	295 (43)	240 (35)	140 (20)	105 (15)
IN-713C	370 (54)	305 (44)	305 (44)	215 (31)	130 (19)	70 (10)
IN-738 C	470 (68)	345 (50)	330 (48)	235 (34)	130 (19)	90 (13)
IN-738 LC	430 (62)	315 (46)	295 (43)	215 (31)	140 (20)	90 (13)
IN-100	455 (66)	365 (53)	360 (52)	260 (38)	160 (23)	90 (13)
MAR-M 247 (MM 0011)	585 (85)	415 (60)	455 (66)	290 (42)	185 (27)	125 (18)
MAR-M 246	525 (76)	435 (62)	440 (63)	290 (42)	195 (28)	125 (18)
MAR-M 246 Hf (MM 006)	530 (77)	425 (62)	425 (62)	285 (41)	205 (30)	130 (19)
MAR-M 200	495 (72)	415 (60)	385 (56)	295 (43)	170 (25)	125 (18)
MAR-M 200 Hf (MM 009)	—	—	—	305 (44)	—	125 (18)
B-1900	510 (74)	380 (55)	385 (56)	250 (36)	180 (26)	110 (16)
René 77	—	—	310 (45)	215 (31.5)	130 (19)	62 (9.0)
René 80	—	—	350 (51)	240 (35)	160 (23)	105 (15)
IN-625	130 (19)	110 (16)	97 (14)	76 (11)	34 (5)	28 (4)
IN-162	505 (73)	370 (54)	340 (49)	255 (37)	165 (24)	110 (16)
IN-731	505 (73)	365 (53)	—	—	165 (24)	105 (15)
IN-792	515 (75)	380 (55)	365 (53)	260 (38)	165 (24)	105 (15)
M-22	515 (75)	385 (56)	395 (57)	285 (41)	200 (29)	130 (19)
MAR-M 421	450 (65)	305 (44)	310 (46)	215 (31)	125 (18)	83 (12)
MAR-M 432	435 (63)	330 (48)	295 (40)	215 (31)	140 (20)	97 (14)
MC-102	195 (28)	145 (21)	145 (21)	105 (15)	—	—
Nimocast 90	160 (23)	110 (17)	125 (18)	83 (12)	—	—
Nimocast 242	110 (16)	83 (12)	90 (13)	59 (8.6)	45 (6.5)	—
Udimet 500	330 (48)	240 (35)	230 (33)	165 (24)	90 (13)	—
Udimet 710	420 (61)	325 (47)	305 (44)	215 (31)	150 (22)	76 (11)
CMSX-2	—	—	—	345 (50)	—	170 (25)
GMR-235	—	—	—	180 (26)	—	75 (11)
IN-939	—	—	—	195 (28)	—	60 (9)
MM 002	—	—	—	305 (44)	—	125 (18)
IN-713 Hf (MM 004)	—	—	—	205 (30)	—	90 (13)
René 125 Hf (MM 005)	—	—	—	305 (44)	—	115 (17)
SEL-15	—	—	—	295 (43)	—	75 (11)
UDM 56	—	—	—	270 (39)	—	125 (18)
Cobalt Base						
HS-21	150 (22)	95 (14)	115 (17)	90 (13)	60 (9)	50 (7)
X-40 (HS-31)	180 (26)	140 (20)	130 (19)	105 (15)	75 (11)	55 (8)
MAR-M 509	270 (39)	225 (33)	200 (29)	140 (20)	115 (17)	90 (13)
FSX-414	150 (22)	115 (17)	110 (16)	85 (12)	55 (8)	35 (5)
WI-52	—	195 (28)	175 (25)	150 (22)	90 (13)	70 (10)

Source: *Metals Handbook Desk Edition,* ASM International, Materials Park, OH 44073-0002, 1999, p. 412.

mechanical properties. Concurrently, the availability of oxygen at high temperatures accelerates the conversion of some of the metal atoms to oxides. Oxidation proceeds much more rapidly at high temperatures than at room or lower temperatures.

2.2 Mechanical Behavior

In the case of short-time tensile properties (yield strength, ultimate strength), the mechanical behavior of metals at higher temperatures is similar to that at room temperature, but with metals becoming weaker as the temperature increases. However, when steady loads below the normal yield or ultimate strength determined in short-time tests are applied for prolonged times at higher temperatures, the situation is different. Figure 1 illustrates the way in which most materials respond to steady extended-time loads at high temperatures.

Owing to the higher temperature, a time-dependent extension (creep) is noticed under load. If the alloy is exposed for a long time, the alloy eventually fractures (ruptures). The degradation process is called creep or, in the event of failure, creep-rupture (sometimes stress-rupture), and alloys are selected on their ability to resist creep and creep-rupture failure. Data for superalloys frequently are provided as the stress that can be sustained for a fixed time (e.g., 100-h rupture) versus the temperature. Figure 2 shows such a plot with ranges of expected performance for various superalloy families. One of the contributory aspects of elevated temperature failure is that metals tend to come apart at the grain boundaries when tested for long times above about 0.5 of their absolute melting temperature. Thus, fine-grained alloys, which are usually favored for lower temperature applications, may not be the best materials for creep-rupture limited applications at high temperatures. Elimination or reorientation of grain boundaries is sometimes a key factor in maximizing the higher temperature life of an alloy.

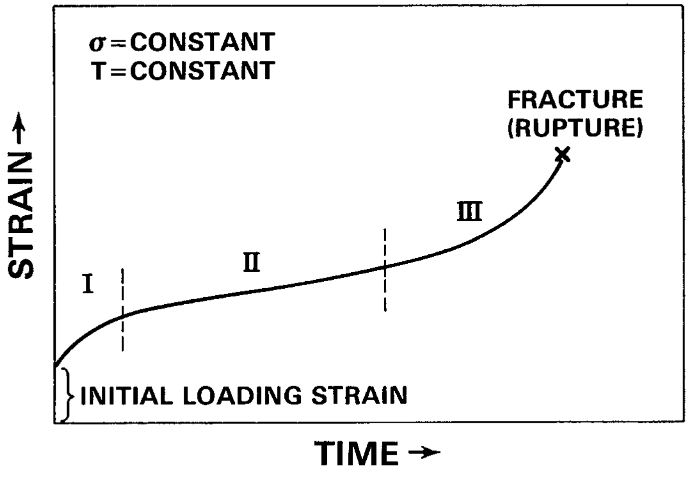


Fig. 1 Creep-rupture schematic showing time-dependent deformation under constant load at constant high temperatures followed by final rupture. (All loads below the short-time yield strength. Roman numerals denote ranges of the creep-rupture curve.)

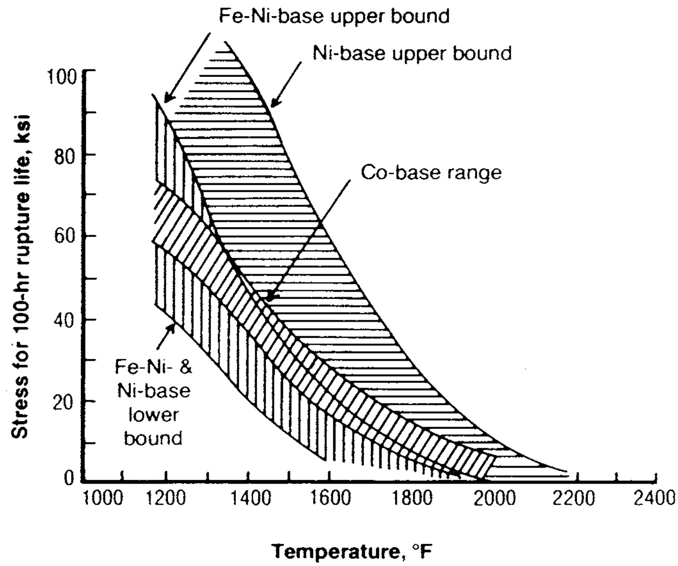
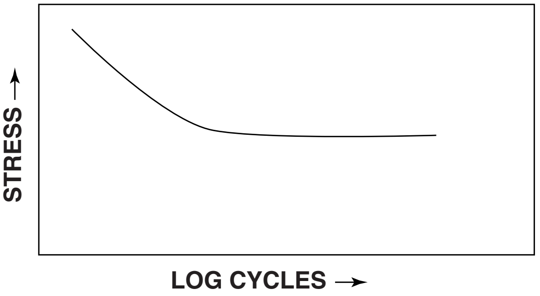


Fig. 2 Creep-rupture curves showing ranges for superalloy families. (From *Superalloys Source Book,* ASM International, Materials Park, OH 44073-0002, 1984, p. 3.)

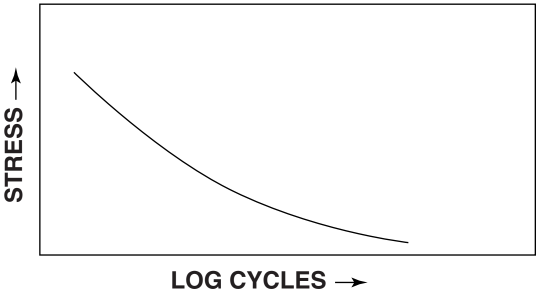
Cyclically applied loads that cause failure (fatigue) at lower temperatures also cause failures in shorter times (lesser cycles) at high temperatures. For example, Fig. 3 shows schematically how the cyclic resistance is degraded at high temperatures when the locus of failure is plotted as stress versus applied cycles (S–N) of load. From the S–N curves shown, it should be clear that there is not necessarily an endurance limit for metals and alloys at high temperatures. Cyclic loads can be induced not only by mechanical loads in a structure, but also by thermal changes. The combination of thermally induced and mechanically induced loads leads to failure in thermal-mechanical fatigue (TMF). TMF failures occur in a relatively low number of cycles. Thus TMF is a low cycle fatigue (LCF) process while lower load mechanical fatigue leads to failure in a high number of cycles (HCF). LCF failures in structures can be mechanically induced or TMF-type. In airfoils in the hot section of gas turbines, TMF is a major concern. In highly mechanically loaded parts, such as gas turbine disks, LCF is the major concern. HCF normally is not a problem with superalloys unless a design error occurs and subjects a component to a high-frequency vibration that forces rapid accumulation of fatigue cycles.

While life under cyclic load (S–N behavior) is a common criterion for design, resistance to crack propagation is an increasingly desired property. Thus, the crack growth rate versus a fracture toughness parameter is required. The parameter in this instance is the stress intensity factor (K) range over an incremental distance which a crack has grown—the difference between the maximum and minimum K in the region of crack length measured. A plot of the resultant type (da/dn vs. ΔK) is shown in Fig. 4 for several wrought superalloys.

The nature of superalloys is that they resist the creep-rupture process better than other materials, have very good higher temperature short-time strength

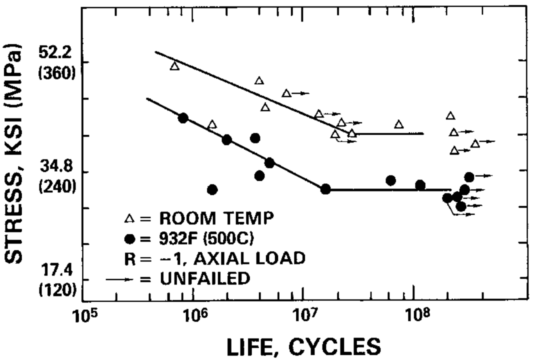


Typical Fatigue Curve at Low Temperature



Typical Fatigue Curve at High Temperature

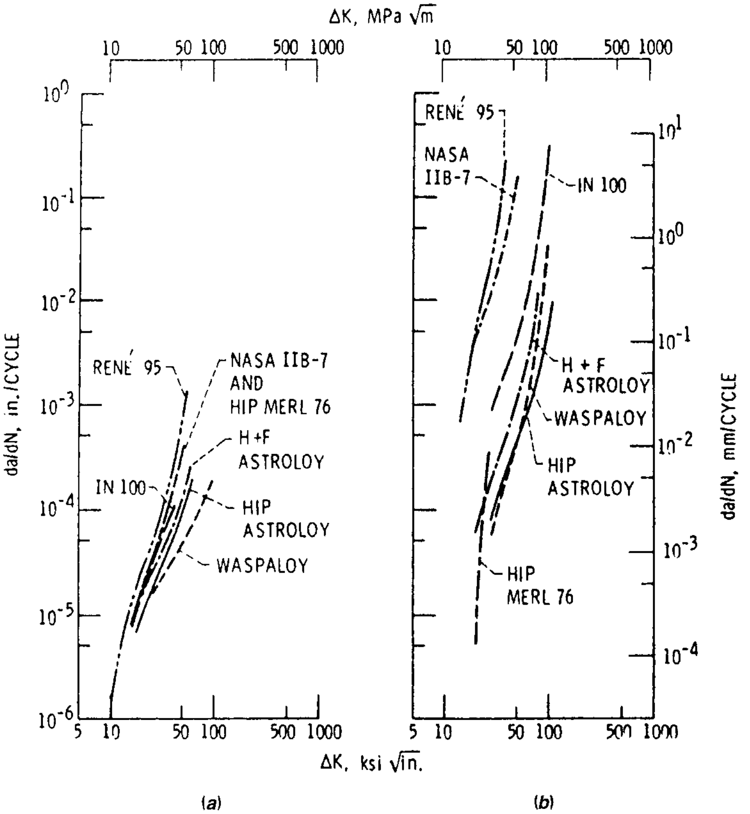
(*a*)



(*b*)

Fig. 3 Stress versus cycles to failure (S–N) curves showing: (*a*) schematic typical fatigue response at high temperature vs. that at lower temperature and (*b*) actual fatigue curves at room and elevated temperature for a specific nickel-base superalloy.

(yield, ultimate), very good fatigue properties (including fatigue crack propagation resistance) and combine these mechanical properties with good to exceptional oxidation resistance. Consequently, superalloys are the obvious choice when structures are to operate at higher temperatures. Generally, the temperature range of superalloy operation is broken up into the intermediate range of about 540°C (1000°F) to 760°C (1400°F) and the high-temperature range that occurs above about 816°C (1500°F).



(*a*) (*b*)

Fig. 4 Crack growth rate (*da*/*dn*) versus toughness change (Δ*K*) curves for several superalloys at 649°C (1200°F). Note that HIP = hot isostatically pressed and H + F = HIP + forge. Unless otherwise noted, all alloys are forged. (From *Superalloys II,* Wiley, 1987, p. 284. Used by permission.)

3 PROPERTIES OF SUPERALLOYS

3.1 Physical/Environmental

Iron, cobalt, and nickel, the bases of superalloys, are transition metals located in a similar area of the periodic table of the elements. The density, melting point, and physical properties of the superalloy base elements are given in Table 10. As can be seen, pure iron has a density of 7.87 g/cm^3 (0.284 lb/in.^{3}) , while pure nickel and cobalt have densities of about 8.9 g/cm^3 (0.322 lb/in.^{3}). The superalloys are created usually by adding significant levels of the alloy elements chromium, aluminum, and titanium, plus appropriate refractory metal elements such as tungsten and molydenum to the base metal. Densities of superalloys are a function of the amounts of these elements in the final compositions. For example, iron-nickel-base superalloys have densities of around 7.9–8.3 g/cm^3 (0.285–0.300 lb/in.^{3}), not too dissimilar to densities of nickel-base superalloys, which may range from about 7.8 to 8.9 g/cm^3 (0.282 to 0.322 lb/in.^{3}). Cobalt-base superalloy densities, on the other hand, range from about 8.3 to 9.4 g/cm^3 (0.300 to 0.340 lb/in.^{3}). Aluminum, titanium, and chromium reduce superalloy

Table 10 Some Physical Properties of Superalloy Base Elements

		Melting Point		Density		Expansion Coefficient[a]		Thermal Conductivity[a]	
	Crystal Structure	°F	°C	lb/in^3	g/cm^3	°F $\times 10^{-6}$	°C $\times 10^{-6}$	Btu/ft^2/hr/°F/in.	cal/cm^2/s/°C/cm
Co	HCP	2723	1493	0.32	8.9	7.0	12.4	464	0.215
Ni	FCC	2647	1452	0.32	8.9	7.4	13.3	610	0.165
Fe	BCC	2798	1535	0.28	7.87	6.7	11.7	493	0.175

[a] At room temperature.

Source: From *Superalloys II,* Wiley, 1987, p. 14.

density whereas the refractory elements such as tungsten, rhenium, and tantalum increase it. Table 5 gives density, melting range, and physical properties of some nickel- and cobalt-base superalloys.

The melting temperatures of the basis superalloy elements are nickel at 1452°C (2647°F), cobalt at 1493°C (2723°F), and iron at 1535°C (2798°F). When metals are alloyed, there is no longer a single melting point for a composition. Instead, alloys melt over a range of temperature. The lowest melting temperature (incipient melting temperature) and melting ranges of superalloys are functions of composition and prior processing. Just as the basis metal is higher melting, so generally are incipient melting temperatures greater for cobalt-base superalloys than for nickel- or iron-nickel-base superalloys. Nickel-base superalloys may show incipient melting at temperatures as low as 1204°C (2200°F). However, advanced nickel-base single-crystal superalloys having limited amounts of melting-point depressants tend to have incipient melting temperatures equal to or in excess of those of cobalt-base superalloys.

The physical properties of electrical conductivity, thermal conductivity, and thermal expansion of superalloys tend to be low (relative to other metal systems). These properties are influenced by the nature of the base metals (transition elements) and the presence of refractory-metal additions.

The corrosion resistance of superalloys depends primarily on the alloying elements added and the environment experienced. Contaminants in the atmosphere can caused unexpectedly high corrosion rates. The superalloys employed at the highest temperatures are coated to increase oxidation/corrosion resistance. More information follows later in this chapter.

3.2 Mechanical

The superalloys are relatively ductile; the ductilities of cobalt-base superalloys generally are less than those of iron-nickel- and nickel-base superalloys. Short-time tensile ductilities as determined by elongation at failure generally range from as low as 10 pct to as high as around 70 pct, but γ'-hardened alloys are in the lower end, usually between about 10 and 40 pct. Creep-rupture ductilities generally are lower than tensile ductilities. At the 760°C (1400°F) tensile ductility minimum area, creep-rupture ductilities of castings have gone below 1.5 pct; however, most current high-strength PC equiaxed cast alloys have rupture ductilities in excess of 2.0 pct. Single crystal directionally solidified (SCDS) superalloy ductilities can vary with orientation of the single crystal relative to the testing direction.

Superalloys typically have dynamic moduli of elasticity in the vicinity of 207 GPa ($30^\circ \times 10^6$ psi), although moduli of specific polycrystalline (PC) equiaxed alloys can vary from 172 to 241 GPa (25 to 35×10^6 psi) at room temperature depending on the alloy system. Processing that leads to directional grain or crystal orientation can result in moduli of about 124 to 310 GPa (about 18 to 45×10^6 psi) depending on the relation of grain or crystal orientation to testing direction. Dynamic measurement of modulus of elasticity at high temperatures is necessary because static modulus is greatly influenced by high temperatures and shows significant reductions over the dynamic value at a common high temperature. Modulus may drop by around 25–30 pct as temperatures increase from room temperature to 871°C (1600°F).

Short-time tensile yield properties of γ'-hardened alloys range from around 550 MPa (80 ksi) to 1380 MPa (200 ksi) at room temperature. Actual values depend on composition and processing (cast vs. wrought). Wrought alloys tend to have the highest values, with the highest hardener content alloys (e.g., Rene 95, IN 100) having the highest strengths. However, strength is a function of grain size and stored energy and alloys such as U630 or IN 718 can be produced with very high yield strengths. Solid-solution-hardened alloys such as the sheet alloy, Hastelloy X, show lower strengths. Ultimate strengths range from around 690 MPa (100 ksi) to 1520 MPa (230 ksi) at room temperature, with γ'-hardened alloys in the high end of the range.

Superalloys tend to show an increase of yield strength from room temperature up to about 760°C (1400°F) and drop off thereafter. This is in contrast to ordinary alloys that tend to continuously decrease in short-time strength as temperatures increase. Ultimate strengths generally do not show this trend. Concurrently, tensile ductility tends to decrease, with a minimum at around 649°C (1200°F). Many published data for alloys do not show any or much of an increase in tensile yield strength over the range to 760°C (1400°F).

The highest tensile properties are found in the finer grain size wrought or powder metallurgy superalloys used in applications at the upper end of the intermediate temperature regime, perhaps to about 760°C (1400°F). The highest creep-rupture properties invariably are found in the coarser grain cast superalloys used in the high-temperature regime. Rupture strengths are a function of the time at which they are to be recorded. The 1000-h rupture stress capability is obviously lower than the 100-h capability. Creep capability also is a function of the amount of creep permitted in a test. For example, the time to 0.5, 1.0, 2.0, and 5.0 pct might each be valuable for design dependent on a component's intended use. It is much more difficult to find this information than it is to find creep-rupture capability information. Handbooks generally do not carry much creep information. Creep-rupture strengths for 100-h failure at 982°C (1800°F) may range from 45 MPa (6.5 ksi) for an older γ'-hardened wrought alloy such as U500 to 205 MPa (30 ksi) for the PC equiaxed cast superalloy Mar-M 246. Columnar grain and single-crystal alloys can be much stronger.

Cyclic properties are not commonly tabulated for superalloys. Properties of interest would be the 10^3–10^5 and 10^6–10^8 cycle fatigue strength capabilities. This could mean stress for a fixed-cyclic life-to-a-particular-sized crack or stress for a fixed-cyclic life-to-fracture for LCF regimes or only stress for a fixed-cyclic life-to-fracture for HCF regimes. Also, crack propagation rates versus toughness parameter (da/dN vs. ΔK) are desired. The life values, when available, lend themselves to tabulation, but the da/dN values are best represented by graphs. LCF strengths are usually related to an alloy's yield strength while HCF strengths are usually related to an alloy's ultimate strength for wrought alloys used at intermediate temperatures. For cast alloys used in the hottest sections of a gas turbine, there appears to be a relation of TMF strength to the creep strength of an alloy for a given alloy form, as for columnar grain directionally solidified (CGCS) nickel-base superalloys.

Superalloys usually are processed to optimize one property in preference to others. The same composition, if used in cast and wrought state, may have different heat treatments applied to the different product forms. Even when a superalloy is used in the same product form, process treatments may be used to optimize one property over others. For example, an alloy such as Waspaloy was made in wrought form for gas turbine disks. By adjustment of processing conditions, principally heat treatment, substantial yield strength improvements were achieved at the expense of creep-rupture strength.

4 EVOLUTION OF SUPERALLOYS

During the first quarter of the twentieth century, chromium was added at various times to cobalt, nickel, and iron. The resulting products were remarkably resistant to atmospheric (moisture-based) environments and to oxidation at high temperatures. By World War II, some of these alloys, including other alloy elements, had come into use for such applications as resistance wires, dental prostheses, cutlery, and furnace and steam turbine components. With the development of the gas turbine engine during the war, the need became apparent for corrosion-resistant materials to operate in demanding mechanical load conditions at high temperatures. At this point, the fledgling superalloy industry began to expand.

By modifying the stainless steels, higher strengths were achieved without the need for special high-temperature strengthening phases. Phases such as η (a nickel–titanium compound) or γ' (a nickel–aluminum compound) had been introduced into the nickel–chromium families of alloys just prior to the war to produce high strength at high temperatures. The increasing temperatures forced alloy developers to include these phases (η and γ') in the iron-base alloys to take the high-temperature strength characteristics beyond those of the modified stainless steels such as 19-9DL. Alloys were invented in Germany and, after modification, made their way to the United States as A-286 or V-57 and are still in use today.

Nevertheless, the need for creep-rupture strength continually increased. Some of this need was met in the early years by adapting a cobalt-base corrosion-resistant alloy (vitallium) for use in aircraft engine superchargers and, later, to airfoils in the hot sections of gas turbines. Similar cobalt-base superalloys are still in use today. However, creep-rupture requirements for aircraft gas turbine applications soon outstripped those of the iron-nickel-base superalloys and the cobalt-base superalloys, and so nickel-base superalloys, modified to provide more of the hardening phase γ', became increasingly used.

4.1 Improvement of Superalloys by Chemistry Control

The production of superalloy components initially consists of some sort of melting process. The melting produces ingots that are either remelted, converted to billet, converted to powder for subsequent consolidation to a component, or investment cast. Remelting is used to produce an ingot that can be processed to billet for forging or to mill forms (e.g., bar stock). Until the start of the second half of the twentieth century, melting of superalloys was conducted in air or under slag environments. The properties of modern superalloys derive principally from the presence of many elements that are reactive with oxygen and so were being lost to some degree in the customary melting and casting processes of the time. When vacuum melting techniques were introduced to commercial production of articles, they were pioneered by superalloys. Vacuum melting enabled the production of superalloys containing higher amounts of the hardeners aluminum and titanium. Furthermore, the concurrent reduction in gases, oxides, and other impurities caused a significant improvement in the ductility of superalloys. With more hardener content, strengths of superalloys began to increase dramatically. Figure 5 shows the improvement in creep-rupture life achieved with the reduction in oxygen content.

5 MELTING AND CASTING PRACTICES

5.1 General Aspects

The development of superalloys as they are employed today is largely a story of the development of modern melting technology. Whether the final product is to be a forging or an investment casting, the essence of a superalloy's ability to create the properties desired hinges on the correct choice and adaptation of melting principles.

Superalloy melting practices may be classified as either primary (the initial melt of elemental materials and/or scrap that sets the composition) or secondary (remelt of a primary melt for the purpose of controlling the solidification structure). The melt type or combination of melt types selected depends upon both the alloy composition, mill form and size desired, properties desired, and sensitivity of the final component to localized inhomogeneity in the alloy.

5.2 Melting and Refining Superalloys

The two most common primary melt practices are argon–oxygen decarburizing treatment of electric arc processed metal (Arc–AOD) and vacuum induction melting (VIM). The two common secondary melt practices are vacuum arc re-

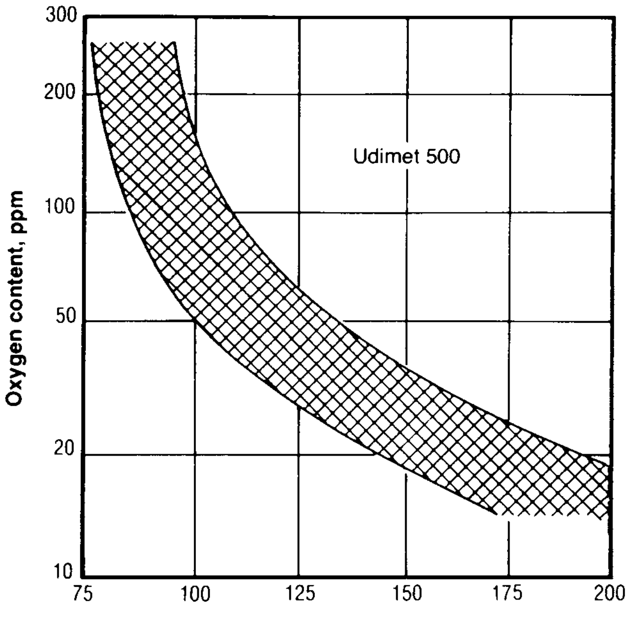


Fig. 5 Improvement of rupture life at 871°C (1600°F) and 172 Mpa (25 ksi) by reduced oxygen content produced by vacuum melting. (From *Superalloys Source Book,* ASM International, Materials Park, OH 44073-0002, 1984, p. 11.)

melting (VAR) and electro slag remelting (ESR). A few alloy mill products may be manufactured from cast ingots after only primary melting. For the majority of superalloys the common practice combinations are Arc-AOD + ESR, VIM + ESR, VIM + VAR, and VIM + ESR + VAR. Common material specifications, such as Aerospace Materials Specifications (AMS), will specify acceptable melting practices.

The Arc-AOD practice used for superalloys is little different from that used for stainless steels. An electric arc furnace is the recipient of the initial charge and uses power from an arc struck between graphite electrodes and the charge to heat and melt the charge. Once the charge melts, no further electric power is needed. Heat is input into the charge by injecting (‘‘blowing’’) oxygen under the surface of the melt to react with elements such as aluminum, titanium, silicon, and carbon. A desulfurizing addition (usually CaO) is made. The slag formed by the oxidation products and the desulfurizing addition is physically removed from the furnace.

The deslagged charge is transferred to the AOD vessel, which is a refractory-lined steel shell with ‘‘tuyeres’’ in the bottom. The tuyeres are used to inject a mixture of argon and oxygen into the molten bath. By controlling the ratio of argon to oxygen, the selective oxidation or reduction of elements may be accomplished. The principal element reduced is carbon. Elements that need to be retained in the melt (particularly chromium) may also be oxidized into the slag. However, additions of aluminum made to the heat will react with the slag and reduce the chromium back into the molten charge. There is no external source of heat to an AOD vessel. The molten charge is heated by making additions of aluminum and oxidizing them. Cooling is accomplished by adding solid scrap to the bath. When the desired composition and pouring temperature are reached, the heat is poured off into a teeming ladle and transferred to the electrode/ingot pouring system. Precautions are taken (shrouding of the pour stream with argon) to minimize the reaction of the pour stream with atmospheric gasses.

The other prevalent primary melt practice is VIM. The VIM furnace consists of a ceramic-lined crucible built up around water-cooled induction coils. The crucible is mounted in a vacuum chamber. The vacuum chamber may have several vacuum ports (of various sizes) built into it so that, without breaking vacuum:

- Charge material may be introduced into the crucible.
- Molds may be introduced into the chamber.
- Systems for removing slag from the pour stream (tundish) may be introduced.
- Sampling of the molten metal may be made for chemistry control.

The charge is separated into three types of components, virgin, reactive, and revert. Note that virgin material is usually elemental material but may also consist of any other type of material that has not been previously vacuum melted. Reactive elements refer to those elements that are strong oxide formers. These are usually the ‘‘hardener’’ elements (titanium, aluminum) in superalloys. Operation of the VIM consists of charging the virgin portion of the charge (minus the reactive portion) into the crucible. The furnace is pumped down (if not already under vacuum) and a measurement made of the increase in pressure (as a function of time) when the vacuum ports are blanked off. This "leak-up rate" is a measurement of the base furnace vacuum integrity. Power is applied to the induction coils. The magnetic fields produced induce a current in the charge material, heating the charge. When the charge is molten, it will evolve gas (refining). The rate of gas evolution is measured by taking leak-up rates. When consecutive stable leak-up rates are obtained, then refining (degassing) is complete. This produces the lowest possible oxygen in the alloy. Reactive additions are then made and revert is added. (Revert is previously vacuum-melted alloy that is of the same or nearly the same composition as the intended melt.)

Additions of some form of calcium are usually made to reduce sulfur in the charge by forming a CaS slag. A chemistry sample is taken and "trim" additions are made to bring the heat to a precise composition. The metal is (top) poured into the molds through a system for metal transfer which also minimizes slag entrainment and regulation of the pour rate. Cast product for direct processing (e.g., for investment casting) is referred to as ingot. Most often, the VIM product is intended for secondary melting and is referred to as an electrode, as both secondary processes consume this product in an electrically heated process.

Compared to VIM, Arc-AOD uses the lowest cost charge material and has higher production rates. That is, Arc-AOD is the lowest cost process. However, Arc-AOD is limited in its ability to precisely control composition in a narrow range (particularly for superalloys with high titanium levels) and also produces much higher oxygen content material. (Note that the choice of secondary melting process may compensate to some degree for the higher oxide inclusion content of the Arc-AOD process.)

Electro slag remelting is the most frequently used secondary melt process for Arc-AOD electrodes. In ESR, alternating current (AC) is applied to an electrode situated inside a water-cooled crucible containing a slag charge. The intended circuit of current is from the electrode, through the molten slag, through the solidifying ingot, through the water-cooled stool, and by symmetrically located buss bars back to the electrode. The slag is generally a CaF_2 base, modified by major additions of CaO, MgO, and Al_2O_3. Minor additions of TiO_2 and ZrO_2 may be made to counteract loss of titanium and zirconium during the melting process. (Note that minor compositional changes may occur during ESR.) The current passing through the slag keeps the slag molten. The molten slag melts the immersed face of the electrode. As the molten metal gathers into drops on the melt surface of the electrode, it is reduced in sulfur (through reaction with the CaF_2) and entrained oxides are incorporated into the slag. The fully formed drop falls through the slag and is collected in the water-cooled crucible. The slag, which has formed a solid layer against the cooled crucible wall, is partially remelted by the molten metal, but remains as a layer between the ingot and the crucible. As the electrode is consumed, an ingot is built up in the crucible. At any given time the ingot contains a pool of molten metal at the ingot top and a zone of liquid + solid between the solid portion of the ingot and the molten pool.

It should be noted that, for a given alloy, there is a critical thickness and angle (to the growth direction) of the liquid + solid zone at which the liquid in that zone may form self-perpetuating channels in the solidification structure. Such channels are known as "freckles" and are of such high solute content that subsequent thermal treatment cannot eliminate the elemental concentration. Thus, the final product may contain channels of hard particles (from the high solute) which are very detrimental to fatigue properties. The more highly alloyed the material being melted, the smaller and shallower must be the liquid + solid zone in order to avoid freckle formation. The shape and thickness of the liquid + solid zone may be modified to be more resistant to freckle formation by reducing the melt rate (the rate of deposition of molten metal into the pool), reducing the ingot diameter or by improving the heat extraction from the sides of the crucible.

Vacuum arc remelting is used in preference to ESR where larger ingot sizes of highly alloyed materials are needed. VAR is a direct current (DC) process conducted in a vacuum. As occurs in ESR, the electrode is melted into a water-cooled crucible. The melting is accomplished by initially striking an arc between the electrode face and the crucible stool. The arc melts the electrode face and the molten metal collects into drops that fall into the crucible to form an ingot against which the arc is maintained. Because there is no additional source of heat (as, e.g., the cap of molten slag in ESR) and no insulating slag skin on the ingot, the profile of the molten zone in VAR is generally shallower (than in ESR) for any given alloy, melt rate, and ingot diameter combination.

There is no compositional change in VAR with the exception of minor elements with high vapor pressures. Thus, residual amounts of detrimental elements such as bismuth and lead may be removed by VAR. Unfortunately, volatile elements such as magnesium (used for control of sulfide shape) are also removed. The removal of magnesium is generally compensated for by providing an elevated level in the primary melt process.

5.3 Pros and Cons of Remelted Ingot Processing

In VAR the interface between the ingot and the crucible does not contain the slag skin found in ESR. Instead, the surface of the ingot, being the first material to solidify, is low in solute elements. More importantly, any oxides or nitrides from the electrode are swept into this surface layer as they melt out of the electrode and onto the surface of the molten pool. This layer is called "shelf." A disruption in the arc stability may cause the arc to undercut a small section of this shelf. If this section drops into the pool, it is unlikely to remelt and thus forms an alloy lean "spot" that contains layers of entrapped oxide and nitride ("dirt"). These inhomogeneous regions are light or "white" etching and are known as "dirty white spots." Their generation is triggered by conditions that do not generally leave any electronic signature (at current levels of detection capability) in the VAR records. While robust VAR process parameters and good electrode quality may minimize the frequency of dirty white spot occurrence, their formation cannot be completely avoided. Thus, the possible presence of dirty white spots in a component (and the degree of their degradation of properties) must be considered in material/melt-process selection for a component.

In selecting ESR as the secondary melt practice, positive considerations are that ESR does not generally form dirty white spots, that ESR products may also have better hot workability than VAR products and that the process yield may be modestly higher (lower cost). Negative considerations are that ESR products cannot be made in large ingot sizes (compared to VAR) without the formation of freckles. The state of the art in ESR controls is such that, at sizes where ESR will normally not form freckles, some undetected abnormality in the melt may cause sufficient disruption that freckles will be produced anyway. ESR is not a robust process (with regard to avoiding defect formation) when operated toward the extreme end of its size capability. However, advances in ESR controls have allowed commercially useful ingots of ESR Waspaloy to be produced while maintaining an adequate distance from the melt regimes where freckle formation would become a concern. More highly alloyed materials (such as IN 718) cannot yet be robustly produced by ESR in similar sizes.

In selecting VAR as the secondary melt practice, positive considerations are that (for a given alloy) VAR will produce a larger ingot (than ESR) without the presence of freckles and that at the selected size the process should be much more robust (in freckle resistance) than is ESR. The principal negative consideration is that dirty white spots will exist in the ingot and that they cannot be detected and their removal assured.

Several producers of critical rotating components in the gas turbine industry have adopted the use of a hybrid secondary melt process: VIM to ensure a low oxygen, precise chemistry initial electrode is followed by ESR. The ESRed electrode will be clean and sound but may contain freckles. After VIM + ESR, the clean, sound electrode is then remelted by VAR. The improved cleanliness and soundness of the electrode facilitates VAR control. This product (referred to as "triple melt") has a much reduced frequency of dirty white spot occurrence compared to double melt (VIM + VAR) product. However, even in triple melt, dirty white spots will occur and must be considered in the component design. The trade-off is the additional cost of an extra processing step versus the enhanced resistance to premature component failure from dirty white spots.

6 COMPONENT PRODUCTION

6.1 Casting Practices to Produce Parts

The principal casting practice is to use investment casting (also known as the lost wax process). A reverse cast model of the desired component is made and wax is solidified in the resultant die. Then a series of these wax models are joined to a central wax pouring stem. The assembly is coated (invested) with appropriate ceramic, processed to remove wax, and fired to strengthen the invested ceramic mold. An alloy is remelted and cast into the mold. Upon solidification, a series of components in the desired form are attached to the central pouring stem. These objects, frequently turbine hot section airfoil components, are removed and then processed to desired dimensions.

Superalloy investment castings now are available in sizes from a few inches in length up to about 48 in. diameter. Turbine airfoils can now be made not only for aircraft gas turbines but also for land-based power turbines with airfoil lengths of several feet.

Conventional investment castings are polycrystalline (PC) equiaxed with more or less randomly oriented grains. Mechanical properties are nominally random but may show some directionality. Increased property strength levels have been achieved by columnar grain directional solidification (CGDS), which removes grain boundaries that are perpendicular to the applied principal load in turbine airfoils. Removal of these grain boundaries dramatically improved longitudinal (parallel to airfoil axis) creep-rupture properties of nickel-base cast superalloys. Transverse boundaries continue to be a problem but their effects are minimized by the addition of hafnium, which enhances grain boundary ductility. The ultimate solution is to directionally solidify a superalloy as a single crystal, i.e., a superalloy with no grain boundaries. Maximum creep-rupture strength in nickel-base superalloys now is achieved with single-crystal directionally solidified (SCDS) alloys.

An interesting benefit of directional solidification in nickel-base superalloys is the reduction of the elastic modulus in the primary solidification direction (parallel to the longitudinal axis). Reduced moduli mean less thermal stress on hot section airfoils and thus longer life in TMF.

Selection of cast superalloys is best conducted in conjunction with an investment casting foundry that can indicate the likelihood of success in casting a given design in the alloy selected. Not all alloys are equally good for casting. Casting defects occur and vary with the alloy composition and casting practice. Traditionally, porosity is a major concern, especially with large castings for the cases of gas turbines. Porosity may be controlled by mold design and pouring modifications. Non-surface-connected porosity may be closed by hot isostatic pressing (HIP) of cast articles. Surface porosity in large castings may be repaired by welding. Other casting concerns have included intergranular attack (IGA) caused during chemical removal of molding materials, selectively located coarse grains in PC materials, misorientation and spurious grains in DS alloys, etc. Alloys may be modified in chemistry to optimize product yield but with a possible compromise in properties. Product yield will be an important determinator of final component cost.

6.2 Forging and Powder Metal Superalloys

Forging is the most common method of producing wrought components for superalloy applications. Mill products such as bar stock, wire, etc. are produced, but the most demanding applications use forged wrought ingot metallurgy components. Disks are forged to near-net size or to approximate shape using large presses and appropriate billets from previously produced ingots. The forging process requires the application of heat and pressure to move the alloy from its billet or powder metallurgy preform stage to a finished disk. One or more intermediate shape stages usually are involved when conventional forging is practiced. Isothermal (usually superplastic) forging may go directly from billet to final stage in one step.

As alloy strengths increased, it became increasingly difficult to move the alloy around during forging. Higher pressures were required and defects became more probable. Superplastic forging (isothermal forging) became available in the time frame when the strengths of superalloys were bumping up against metallurgical limits. In addition, the discipline of powder metallurgy opened the way to create billets of the highest strength alloys without the positive segregation defects (of alloy elements) that were making casting and forging of billets to final parts so difficult.

In powder metallurgy, used principally for alloys such as Rene 95, IN 100 or other advanced alloys, a preliminary ingot is remelted and then atomized to small droplets that solidify with limited or no segregation of alloy elements. The powders thus produced are subsequently compacted together by HIP or by extrusion and processed to achieve 100-pct density. The aggregates are homogenous and display uniform properties. Sometimes the powder is pressed to form the final size/shape directly, but usually the powder is compacted to an intermediate stage (e.g., extruded billet) and isothermally forged to final form. Powder metallurgy is expensive, but the savings in subsequent machining costs and the ability to control defects make a reasonable cost trade. Possible defects in PM include carbide stringers and ceramic inclusions from the powder production process.

Conventional alloys such as Waspaloy, U 720, and IN 718 are produced by ingot metallurgy and standard forging practices. High-strength alloys such as Rene 95, IN 100, and Rene 88 generally are produced using powder metallurgy techniques.

6.3 Joining

Components of superalloys often could be advantageously produced or repaired by joining processes in the fledgling days of the superalloy industry. That is not necessarily true today. Cobalt-base superalloys, which do not depend on precipitated phases for strength generally are amenable to fusion welding. Sheet metal structures have been fabricated and repair welding is routinely practiced on cobalt-base superalloys. Wrought solution-hardened iron-nickel-base superalloys can be welded, but most precipitation-hardened versions are done with difficulty. Precipitation-hardened alloys tend to crack on fusion welding and properties of many precipitation-hardened alloys are degraded by the heat of the joining processes. These same concepts apply to the precipitation-hardened nickel-base superalloys, which become increasingly more difficult to weld as the amount of hardener in an alloy increases. An exception is the nickel-base (sometimes called nickel-iron-base) superalloy IN 718 which uses large amounts of niobium to harden the alloy. Figure 6 shows the relationship between hardener content and weldability for nickel-base superalloys.

While inertia bonding (nonfusion process) has been used to join nickel-base superalloys, conventional fusion welding is not customarily used successfully although electron beam welding may be useful. Other solid-state joining processes such as transient liquid-phase (TLP) bonding have been used with some measure of success.

The essence of the joining situation for superalloys is that nickel-base airfoils are not repair welded if cracks appear. Cobalt-base airfoils, on the contrary, may be welded to extend life. Sheet metal of cobalt-base, solid-solution-strengthened nickel-base and of lower hardener content nickel-base alloys can be joined and repair welded, as can iron-nickel-base alloys. The nickel-base alloy of choice for components requiring welding is IN 718, which, owing to the unique aspects of its hardening by γ'', can be fusion welded with relative ease.

6.4 Summary of Manufacturing Process

Figure 7 presents a view of the superalloy manufacturing process. Many producers have been involved in the business of producing superalloys over the

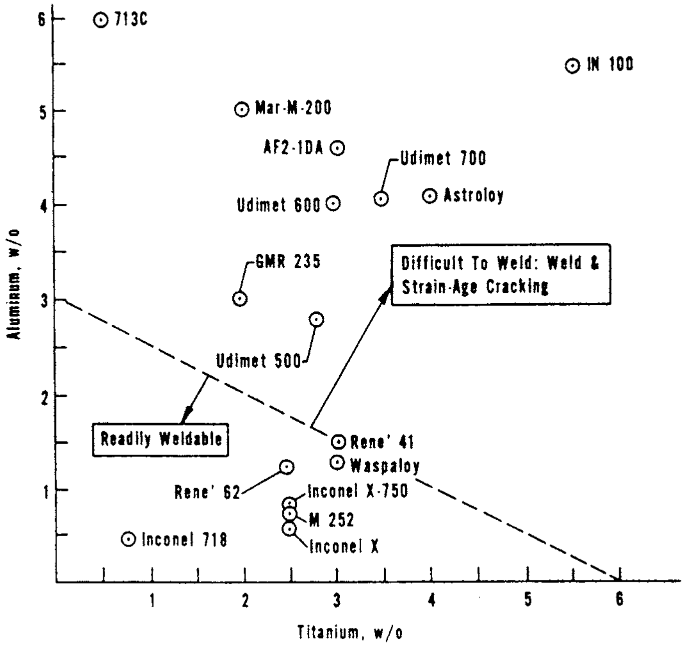


Fig. 6 Weldability diagram for some γ'-strengthened nickel-base superalloys showing the influence of total age hardening elements (Al plus Ti) on cracking tendency. (From *Superalloys II,* Wiley, 1987, p. 152. Used by permission.)

years. During the past 50 years of progress, many advances have been made. New companies have formed and others gone out of business. Following are some current sources of superalloy expertise in ingot melting, component forging, and article casting:

General Information

ASM International, Materials Park, OH; www.asminternational.org

Nickel Development Institute, Toronto, ON, Canada; www.nidi.org

Cobalt Development Institute, Guildford Surrey, UK; www.cobaltdevinstitute.com

International Chromium Development Institute, Paris, France; www.chromium-assoc.com

Specialty Steel Industry of North America, Washington, DC; www.ssina.com

The Minerals, Metals & Materials Society, American Institute of Mining, Metallurgical and Petroleum Engineers New York, NY; www.tms.org

Melting/Ingot Production, Forming and/or Mill Products

Special Metals Corporation with locations at New Hartford, NY, Huntington, WV, and Hereford, UK; www.specialmetals.com

Carpenter Technology Corporation, Wyomissing, PA; www.cartech.com

Haynes International, Kokomo, IN; www.haynesintl.com

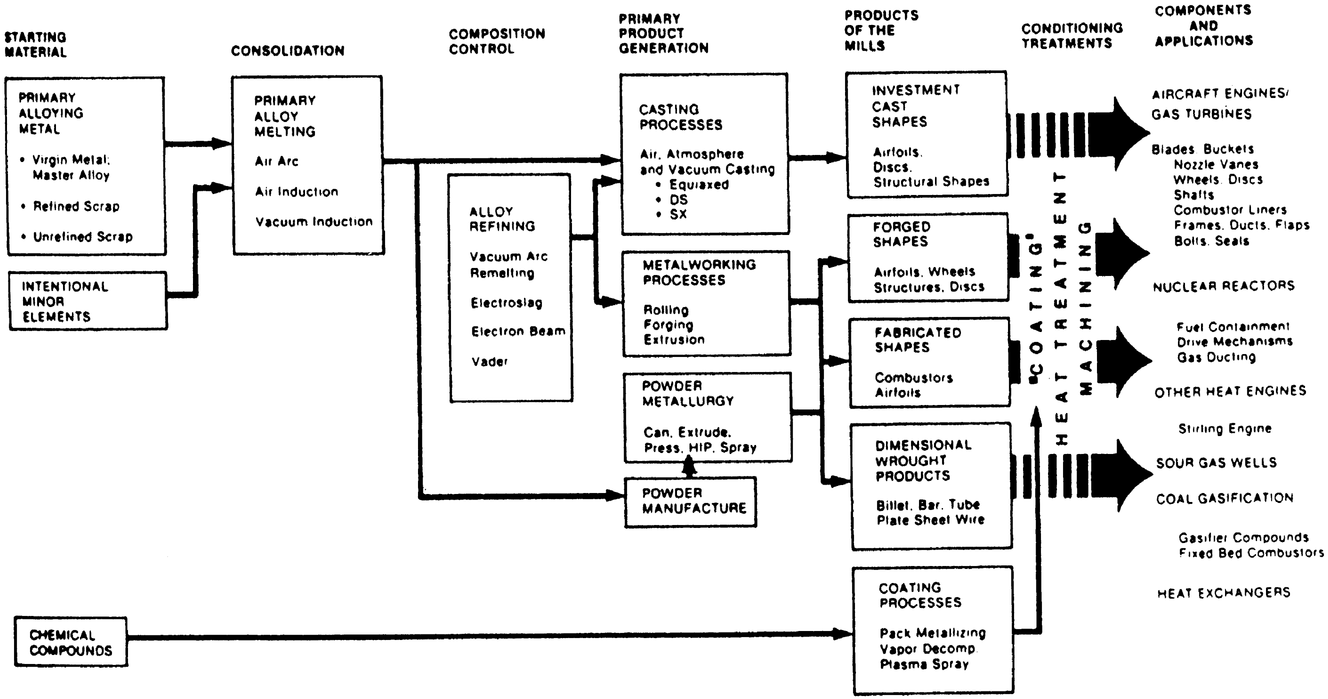


Fig. 7 View of the superalloy manufacturing process. (From *Superalloys II,* Wiley, 1987, p. 23. Used by permission.)

Teledyne Allvac, An Allegheny Technologies Co., Monroe, NC; www.allvac.com

Cannon-Muskegon Corp., Subsidiary of SPS Technologies, Muskegon, MI; www.greenvillemetals.com/cmgroup.htm

Howmet with locations worldwide, including Dover, NJ, and Devon, UK; www.howmet.com

Doncasters PLC with locations including Sheffield, UK; www.doncasters-bramah.com

Precision Rolled Products, Florham Park, NJ; No web site

Investment Castings

Howmet Corp. with locations including Hampton, VA, Whitehall, MI, LaPorte, IN, Wichita Falls, TX, and Devon, England, Gennevilliers, France, Dives, France, and Terai, Japan; www.howmet.com

Precision Cast Parts with locations including Minerva, OH, Cleveland, OH, Mentor, OH, and Douglas, CA; www.precast.com

Doncasters Precision Castings with locations including Droitwich, Worcs, UK; www.doncasters-deritend.com; Bochum, Germany; www.doncasters-bochum.com, and Groton, CT; www.doncasters.com

Hitchiner Manufacturing Co., Inc. Gas Turbine Div., Milford, NH; www.hitchiner.com

Forgings

Wyman Gordon Co. with locations including N. Grafton, MA, Livingston, Scotland, and Houston, TX; www.wyman-gordon.com

Schlosser Forge, Cucamonga, CA; www.aerospace-engine-parts.com

Ladish Co, Inc., Cudahy, WI; www.ladish.com

Carlton Forge Works, Paramount, CA; website unknown

Carmel Forge, Tirat Carmel, Israel; website unknown

Doncasters PLC with locations including Monk Bridge, UK; www.doncasters-monkbridge.com, Blaenavon, UK; www.doncasters-blaenavon.com, and Leeds, UK; www.doncasters.com

Thyssen Umformtechnik, Remscheid, Germany; www.tut-gmbh.com Fortech, Clermont–Ferrand, France; website unknown

Firth Rixson with locations including Monroe, NY, and Verdi, NV; website unknown

Forged Metals, Fontana, CA; website unknown

Coating and/or Refurbishment/Repair

Chromalloy Gas Turbine Corp. with locations including Carson City, NV, Gardena, CA, Orangeburg, NY, Harrisburg, PA, Middletown, NY, Columbus, IN, Manchester, CT, and Phoenix, AZ; websites including www.chromalloy-cnv.com, www.chromalloy-cla.com, and www.chromalloyhit.com

Sermatech International Inc. with locations including Limerick, PA, Muncie, IN, Houston, TX, and Manchester, CT; www.sermatech.com

This list almost certainly will change and only should be used as a guide to locate potential producers. Most consumers deal with the forging and investment casting vendors who produce the product for subsequent final processing. For best control of properties, many consumers maintain liaison with melters and frequently audit all aspects of the manufacturing process. It is vital to remember in superalloy selection that most superalloys are not off-the-shelf consumer items. They are made to user specifications that may vary from user to user and time to time. Diligence in working with the producers will pay dividends in optimum properties and fewer difficulties for users of superalloys. This concept is indigenous to the industry and superalloy users for mission-critical or human-life-critical applications. It is more costly than buying off-the-shelf. For some applications such as in oil development or coal gasification, there may be less stringent controls that permit off-the-shelf purchase of mill products. No forging or investment casting is an off-the-shelf item.

7 OTHER ASPECTS OF SUPERALLOY SELECTION

7.1 Corrosion and Coatings for Protection

Initial superalloys were intended to achieve strength with adequate oxidation resistance, and this was accomplished with superalloys that contained upward of 20 pct chromium. Oxidation resistance up to temperatures around 982°C (1800°F) was excellent. However, to increase the design flexibility of nickel-base superalloys, chromium content was reduced to permit more hardener to be added. Concurrently, some superalloys were put into service with environments (e.g., marine salts) that intensified oxidation or ion-induced attack such as hot corrosion. Also, the operating temperatures (surface environment temperatures) to which the alloys are exposed in the most demanding high-temperature conditions increased with the strength increases of the available alloys.

At higher temperatures, the chromium oxide formed during prior heat treatment was less protective and did not regenerate with exposure to high temperatures. General oxidation and intergranular oxidation along the grain boundaries of superalloys began to be a problem with chromium-protected superalloys, However, the problem was not as great as anticipated owing to the protective nature of aluminum oxide formed in greater amounts by the higher aluminum values of second- and third-generation γ'-hardened superalloys. Nevertheless, general oxidation occurred and still reduced cross sections, thus effectively increasing stresses in the remaining material. The grain boundary oxidation created notches. The combination of these events was of concern, and to protect against them, coatings were applied to superalloys.

The early coatings were diffusion coatings produced by pack aluminizing or slurry application. Chemistry of the coating was determined by the chemistry of the alloy. Later coatings were produced by overlaying a specific chemistry of a protective nature on the surface of the component using an appropriate physical vapor deposition unit. The diffusion-type coatings can coat internal (non-line-of-sight) surfaces, while the overlay coatings can only coat external surfaces that can be seen by the coating apparatus. The diffusion coatings are cheaper and, for a given thickness, probably nearly as protective as the overlay coatings. However, the overlay coatings can be made nearly twice as thick as the diffusion coatings, so overlay coatings often are the coatings of choice for turbine airfoil applications. Diffusion coatings are used to coat internal cooling passages in hot-section airfoils. Some commercial diffusion coating processes are available but virtually all overlay coating processes have been developed by superalloy users such as the aircraft gas turbine manufacturers.

Overlay coatings are generally more expensive than diffusion coatings. Some diffusion coatings are deposited in conjunction with precious metals such as gold or platinum. There are significant benefits to this incorporation of the noble metals in the coating but costs do go up.

Coating and corrosion technology are complex and do not lend themselves to a simple overall description and formula for protection. Hot corrosion phenomena are found below about 927°C (1700°F). Coatings and higher chromium content of an alloy act to inhibit surface attack. Coatings, in general, preserve the surface so that a component may be reused by removing and then restoring the coating.

Coating selection will be based on knowledge of oxidation/corrosion behavior in laboratory, pilot-plant, and field tests. Attributes that probably will be required for successful coating selection include:

- High resistance to oxidation and/or hot corrosion
- Ductility sufficient to provide adequate resistance to TMF
- Compatibility with the base alloys
- Low rate of interdiffusion with the base alloy
- Ease of application and low cost relative to improvement in component life
- Ability to be stripped and reapplied without significant reduction of base-metal dimensions or degradation of base-metal properties

7.2 Special Alloys for Hot-Corrosion Resistance

As temperature decreases below about 927°C (1700°F), the amount of hot corrosion attack decreases. At temperatures below around 760°C (1400°F), under certain conditions the hot corrosion mechanism changes. Attack may begin to increase dramatically with decreasing temperature but then decreases once more as temperatures drop below about 649°C (1200°F). In this lower temperature regime, the province of land-based power gas turbines, attack is resisted best by producing chromium oxide on the surface. Consequently, alloys for this region and application are those such as IN 738 specifically designed to have higher chromium levels, sometimes back up above 20 pct. (IN 939 is an alloy in this latter category.) Other alloys were devised for optimum hot corrosion resistance in higher temperature service. Rene 80 and IN 792 are such alloys.

7.3 Thermal Barrier Coatings

Allied with superalloy protective coating technology is the development of ceramic, so-called thermal barrier coatings (TBC). TBCs aim to reduce a superalloy's surface temperature by several hundred degrees, enabling a superalloy to operate at lower temperatures in a region where the superalloy may have higher strength. TBCs have found use and can be tailored to operate on a wide range of alloys. TBCs make use of overlay coating technology. A thin overlay coat serves as the bond interface between the ceramic of the TBC and the base superalloy.

8 ALLOY SELECTION SUMMARY

8.1 Intermediate-Temperature Applications

Available Alloys

Wrought alloys generally are used. Waspaloy and Astroloy were standard nickel-base superalloys selected in the past. Waspaloy is available but Astroloy may not be as readily procured. U-720 has found many applications.

Castings may be used for some applications of significant physical size. Large case structures for gas turbines are routinely cast in IN 718. However, in most instances, wrought alloys generally are used. Ductilities and fracture toughness of wrought alloys are better than those of cast alloys. Strength in tensile tests usually is better too. Creep may be of concern but rupture life normally is not an issue. Iron–nickel alloys such as A-286 might be considered as they may have sufficient strength and will be considerably less costly than their nickel-base counterparts. The best alloy to consider is the nickel-base (sometimes called nickel-iron-base) superalloy IN 718.

Superalloy IN 718 is the most widely used superalloy today. As a wrought alloy, it can be made in various strength levels and is the most weldable high-strength superalloy available. IN 718's weldability is a significant factor in its application as large cast cases.

Costs of IN 718 are lower than some other superalloys because of the widespread use of the alloy. IN 718 also is unique in that it contains none of the strategic element cobalt. IN 718 received a significant application boost in the late 1970s when a cobalt "shortage" caused the price of cobalt to soar to astronomical levels. IN 718 at that time faced continued competition from alloys such as Waspaloy and Astroloy. The lack of cobalt in the alloy swung the pendulum to IN 718 and in the succeeding 20 years, IN 718 has solidified its position as the most widely used superalloy.

In lieu of IN 718, alloys such as Waspaloy, U-720, and others can be adapted to designs. Powder metal techniques allow IN 100, Rene 95, and other high-strength and damage-tolerant alloys to be fabricated and used. An important trend in the intermediate temperature area is the development of dual material/property gas turbine disks. One of the major concerns for gas turbine disks is the difficulty of getting all desired properties in one material. Extensive work has been done to validate the position that a disk may be made of more than one material. Demonstrations have shown that creep-resistant rims can be attached to different core materials which emphasize brute-strength and fatigue resistance. Selection of such materials for an application, however, would require extensive alloy/process development.

What to Look for in Wrought Alloys

If alloys to be used are intended for massive applications such as turbine disks, high tensile yield and ultimate strength are desired. Good tensile ductility is important and good mechanical LCF behavior with acceptable crack propagation rates at expected load is a must. If an alloy is to be used as sheet, then good formability is a must, coupled with good weldability. Massive parts such as disks can benefit from good forgeability, but such a quality generally does not exist when high tensile strengths are required. Powder metallurgy processing enables production of forged components not otherwise processable. Inspectability of sonic finished shapes is crucial. Cost is a very important factor, but one that may have to be subverted to properties and processing if a desired component is to be made. Of course, use of special processing techniques such as powder metallurgy may enable a part to be formed that could not be made in any other way and so a high cost may be worth paying.

The essence of superalloy selection for intermediate temperature applications is that there are conventional alloys of capability similar to Waspaloy and down that can be procured and forged by conventional means. Similarly, sheet alloys are available that can be manipulated in conventional ways. For the higher strength applications, there are no easy off-the-shelf technologies or alloys that can just be picked from a catalog and put to work. Selection of alloys is a preliminary step that must be expanded upon to get data and components in a reasonable time frame at acceptable costs.

8.2 High-Temperature Applications

Available Alloys

The highest temperature applications invariably require cast superalloys where the requirement of maximum high-temperature strength is concerned. Some applications, such as combustion chambers, may require less strength and sheet alloys are applied. Nickel- and cobalt-base sheet is available. Hastelloy X, IN 617, HA 188, and others have long experience records. HA 230 has been used extensively. Property data generally are available for these alloys. The material purchased is a mill product and normally is readily available.

Turbine airfoil alloys or some combustor nozzles require stronger alloys than the old standby cobalt-base and early nickel-base PC cast alloys. Many of these highest strength alloys are proprietary to various companies, usually aircraft engine providers. The strongest alloys are the single-crystal nickel-base superalloys.

Cast superalloys for the bottom end of the high-temperature application spectrum may be those standbys such as IN 713 or even cobalt-base alloys such as X-40, which have decades of experience. IN 713 is a good general-purpose PC equiaxed casting alloy that also happens to have no cobalt. Alloys for higher temperatures include U-700, Rene 80, IN 792, IN 100 (Rene 100), Mar-M 246, Mar-M 247, Mar-M 200CG (PWA 1422), Rene 125, PWA 1480, Rene N4, and CMSX alloys such as CMSX-6. Some alloys (PWA 1422, Rene 125) are used only as CGDS components and others only as SCDS components (PWA 1480, Rene N4, most CMSX alloys). Alloys such as Rene 80 have been available in CGDS as well as PC equiaxed form. Others such as IN 792 have been available not only as PC equiaxed components but also in single-crystal form. A defining feature of the short list above is that nearly half of the alloys mentioned are associated with specific aircraft gas turbine companies. Although non-U.S. alloys were omitted from the list, some non-U.S. alloys also tend to be associated with specific manufacturing company ownership. Thus, many of the alloys that satisfy the most demanding environments may not be available for applications outside of their corporate patent realm.

What to Look for in Cast Alloys

If alloys are to be used for turbine airfoils, high creep and creep-rupture strengths are required. To maximize strength, the alloys for the most demanding applications in high-pressure turbine (HPT) sections should be SCDS materials. In addition to maximizing creep-rupture strength, TMF strength is optimized by the reduction in modulus achieved by orienting a specific direction of the superalloy crystal parallel to the airfoil axis. An alloy for the most stringent turbine airfoil applications will have a high melting point and good to excellent oxidation resistance, the ability to accept a coating and good LCF strength at temperatures where the airfoil attachment is made to a disk. These temperatures are around 760°C (1400°F). SCDS processing will also ensure that thin section properties are optimized. As section thickness is reduced, for a fixed load, a superalloy ruptures in less time than a standard thick test bar would fail. The order of property reduction is PC equiaxed, most reduced; CGDS, less; and SCDS, least.

For turbine vanes where no centrifugal load exists, airfoils may be made from PC equiaxed high-strength cast cobalt-base alloys instead of DS processed nickel-base alloys. High incipient melting temperature is desired for first-stage turbine vanes. A special type of superalloy, an oxide dispersion-strengthened (ODS) alloy has been used for turbine vanes in some applications. One such alloy, MA 754, relies on yttria dispersed in a corrosion-resistant nickel–chromium matrix to provide adequate creep-rupture capability. ODS alloys are not common. MA 6000 is another such alloy that may have enough strength for a high-pressure turbine blade in aircraft gas turbines. A problem with PC equiaxed airfoils is that the thermal mechanical stresses are much higher than on CGDS or SCDS parts owing to the higher modulus of PC equiaxed parts. The modulus of the CGDS and SCDS parts may be only 60 pct of the value for the PC equiaxed nickel-base cast alloys. In the most demanding conditions, TMF problems must be minimized by using oriented grain or crystal structures to reduce stresses.

For low-pressure turbine (LPT) airfoils, alloys such as the IN 100 (Rene 100) or IN 792 and Rene 80 PC equiaxed alloys previously used for HPT airfoils may be chosen. If temperatures or stress conditions are sufficiently relaxed, IN 713, U700, or similar first-generation PC equiaxed cast nickel-base superalloys may suffice.

9 FINAL COMMENTS

Many superalloys are available but not so many have been adopted for use. A principal reason for this situation is the high cost of proving the worth and safety of a new material. Within most aircraft engine companies, only a few airfoil alloys and a comparably short list of disk alloys have ever made it to production. Admittedly this list differs from company to company but the message is the same. If an existing alloy works and a new alloy does not offer some benefit that overrides the development cost of proving up the alloy for its new use, do not change alloys.

If an alloy selector is starting from scratch to pick an alloy for an application, then any commercially available alloy may be fair game. On the other hand, the best alloy may not be available owing to corporate patent protection. Then, selection of another alloy from a superalloy producer may be necessary but may possibly require extensive development costs to get the product in workable form and to determine design properties. If possible, select a known alloy that has more than one supplier and more than one casting or forging source. Work with the suppliers and others in the manufacturing chain to acquire typical or design properties for the alloy in the form it will be used. Generic alloys owned by superalloy melters or developers are best for the alloy selector not associated with one of the big corporate users of superalloys. Companies with proprietary interests usually have nothing to benefit from giving up a technological advantage by sharing design data or even granting a production release to use a proprietary alloy.

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CHAPTER 11

PLASTICS: THERMOPLASTICS, THERMOSETS, AND ELASTOMERS

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Plastics, or polymers, are ubiquitous. Through human ingenuity and necessity natural polymers have been modified to improve their utility and synthetic polymers developed. Synthetic polymers in the form of plastics, fibers, elastomers, adhesives, and coatings have come on the scene as the result of a continual search for man-made substances that can perform better or can be produced at a lower cost than natural materials such as wood, glass, and metal, which require mining, refining, processing, milling, and machining. The use of plastics can

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also increase productivity by producing finished parts and consolidating parts. For example, an item made of several metal parts that require separate fabrication and assembly can often be consolidated to one or two plastic parts. Such increases in productivity have led to fantastic growth in macromolecules. Indeed, the use of plastics has increased almost 20-fold in the last 30 years.

Plastics can be classified in various ways. The two major classifications are thermosetting materials and thermoplastic materials. As the name implies, thermosetting plastics or thermosets are set, cured, or hardened into a permanent shape.[1] The curing, which usually occurs rapidly under heat or ultraviolet (UV) light leads to an irreversible cross-linking of the polymer. Thermoplastics differ from thermosetting materials in that they do not set or cure under heat.[2] When heated, thermoplastics merely soften to a mobile, flowable state where they can be shaped into useful objects. Upon cooling, thermoplastics harden and hold their shape. Thermoplastics can be repeatedly softened by heat and shaped.

Thermoplastics can be classified as amorphous or semicrystalline plastics.[2,3] Most polymers are either completely amorphous or have an amorphous component even if they are crystalline. Amorphous polymers are hard, rigid glasses below a sharply defined temperature, which is known as the glass transition temperature. Above the glass transition temperature the amorphous polymer becomes soft and flexible and can be shaped. Mechanical properties show profound changes near the glass transition temperature. Many polymers are not completely amorphous but are semicrystalline. Crystalline polymers have melting points that are above their glass transition temperature. The degree of crystallinity and the morphology of the crystalline phase have an important effect on mechanical properties. Crystalline plastics will become less rigid above their glass transition temperature but will not flow until the temperature is above the crystalline melting point. At ambient temperatures crystalline/semicrystalline plastics have greater rigidity, hardness, density, lubricity, creep resistance, and solvent resistance than amorphous plastics.

Another classification from cost and performance standpoint polymers can be either commodity or engineering plastics.

Another important class of polymeric resins is elastomers. Elastomeric materials are rubberlike polymers with glass transition temperatures below room temperatures. Below that glass transition temperature an elastomer will become rigid and lose its rubbery characteristics.

1 COMMODITY THERMOPLASTICS

The commodity thermoplastics include polyolefins and side-chain-substituted vinyl polymers.[4–6]

1.1 Polyethylene

Polyethylene (PE) plastics have the largest volume use of any plastics. They are prepared by the catalytic polymerization of ethylene.[7] Depending on the mode of polymerization, there can be three basic types of polyethylene: high-density (HDPE), low-density polyethylene (LDPE) polymer, and linear low-density polyethylene (LLDPE). LDPE is prepared under more vigorous conditions, which result in short-chain branching. LLDPE is prepared by introducing short-branching via copolymerization with a small amount of long-chain olefin.

Table 1 Typical Property Values for Polyethylenes

Property	HDPE	LLDPE/LDPE
Density (mg/m^3)	0.96–0.97	0.90–0.93
Tensile modulus (GPa)	0.76–1.0	—
Tensile strength (MPa)	25–32	4–20
Elongation at break (%)	500–700	275–600
Flexural modulus (GPa)	0.8–1.0	0.2–0.4
Vicat soft point (°C)	120–129	80–98
Brittle temperature (°C)	-100 to -70	-85 to -35
Hardness (Shore)	D60–D69	D45–D55
Dielectric constant (10^6 Hz)	—	2.3
Dielectric strength (MV/m)	—	9–21
Dissipation factor (10^6 Hz)	—	0.0002
Linear mold shrinkage (in./in.)	0.007–0.009	0.015–0.035

Polyethylene polymers are crystalline thermoplastics that exhibit toughness, near-zero moisture absorption, excellent chemical resistance, excellent electrical insulating properties, low coefficient of friction, and ease of processing. Their heat deflection temperatures are reasonable but not high. The branching in LLDPE and LDPE decreases the crystallinity. HDPE exhibits greater stiffness, rigidity, improved heat resistance, and increased resistance to permeability than LDPE and LLDPE.

Specialty grades of PE include very low density (VLDPE), medium density (MDPE), and ultrahigh-molecular-weight PE (UHMWPE).

Some typical properties of PEs are listed in Table 1.

Uses. HDPEs major use is in blow-molded bottles, shipping drums, carboys, automotive gasoline tanks; injection-molded material-handling pallets, crates, totes, trash and garbage containers; household and automotive parts; and extruded pipe products (corrugated, irrigation, sewer/industrial and gas distribution pipes).

LDPE/LLDPEs find major applications in film form for food packaging as a vapor barrier film that include stretch and shrink wrap; for plastic bags such as grocery bags, laundry and dry cleaning bags; for extruded wire and cable insulation; and for bottles, closures and toys.

1.2 Polypropylene

Polypropylene (PP) is prepared by the catalyzed polymerization of propylene.[8,9] PP is a highly crystalline thermoplastic that exhibits low density, rigidity, and good chemical resistance to hydrocarbons, alcohols and oxidizing agents, negligible water absorption, excellent chemical properties, and excellent impact/stiffness balance. Its properties appear in Table 2.

Uses. End uses for PP are in blow molding bottles and automotive parts; injection-molded closures, appliances, housewares, automotive parts, and toys. PP can be extruded into fibers and filaments for use in carpets, rugs, and cordage.

Table 2 Typical Property Values for Polypropylenes

Density (mg/m^3)	0.09–0.93
Tensile modulus (GPa)	1.8
Tensile strength (MPa)	37
Elongation at break (%)	10–60
Heat deflection temperature at 0.45 MPa (°C)	100–105
Heat deflection temperature at 1.81 MPa (°C)	60–65
Vicat soft point (°C)	130–148
Linear thermal expansion (mm/mm·K)	3.8×10^{-5}
Hardness (Shore)	D76
Volume resistivity (Ω·cm)	1.0×10^{17}
Linear mold shrinkage (in./in.)	0.01–0.02

1.3 Polystyrene

Catalytic polymerization of styrene yields general-purpose polystyrene (GP PS) often called crystal polystyrene. It is a clear, amorphous polymer that exhibits high stiffness, good dimensional stability, moderately high heat deflection temperature, and excellent electrical insulating properties. However, it is brittle under impact and exhibits very poor resistance to surfactants and solvents. Its properties appear in Table 3.

Uses. Ease of processing, rigidity, clarity, and low cost combine to support applications in toys, displays, consumer goods, and housewares such as food packaging, audio/video consumer electronics, office equipment, and medical devices. PS foams can readily be prepared and are characterized by excellent low thermal conductivity, high strength-to-weight ratio, low water absorption, and excellent energy absorption. These attributes have made PS foam of special interest as insulation boards for construction, protective packaging materials, insulated drinking cups, and flotation devices.

1.4 Impact Polystyrene

Copolymerization of styrene with a rubber, polybutadiene, can reduce brittleness of PS, but only at the expense of rigidity and heat deflection temperature. Depending on the levels of rubber, impact polystyrene (IPS) or high-impact polystyrene (HIPS) can be prepared. These materials are translucent to opaque and

Table 3 Typical Properties of Styrene Thermoplastics

Property	PS	SAN	IPS/HIPS	ABS
Density (mg/m^3)	1.050	1.080	1.02–1.04	1.05–1.07
Tensile modulus (GPa)	2.76–3.1	3.4–3.9	2.0–2.4	2.5–2.7
Tensile strength (MPa)	41–52	65–76	26–40	36–40
Elongation at break (%)	1.5–2.5	—	—	15–25
Heat deflection temperature at 1.81 MPa (°C)	82–93	100–105	80–87	80–95
Vicat soft point (°C)	98–107	110	88–101	90–100
Notched Izod (kJ/m)	0.02	0.02	0.1–0.3	0.1–0.5
Linear thermal expansion (10^{-5} mm/mm·K)	5–7	6.4–6.7	7.0–7.5	7.5–9.5
Hardness (Rockwell)	M60–M75	M80–M83	M45, L55	R69–R115
Linear mold shrinkage (in./in.)	0.007	0.003–0.004	0.007	0.0055

generally exhibit poor weathering characteristics. Typical properties appear in Table 3.

1.5 SAN (Styrene/Acrylonitrile Copolymer)

Copolymerization of styrene with a moderate amount of acrylonitrile provides a clear, amorphous polymer (SAN) with increased heat defection temperature and chemical resistance compared to polystyrene. However, impact resistance is still poor. Typical properties appear in Table 3.

Uses. SAN is utilized in typical PS-type applications where a slight increase in heat deflection temperature and/or chemical resistance is needed, such as housewares and appliances.

1.6 ABS

ABS is a ter-polymer prepared from the combination of acrylonitrile, butadiene, and styrene monomers.[10] Compared to PS, ABS exhibits good impact strength, improved chemical resistance, and similar heat deflection temperature. ABS is also opaque. Properties are a function of the ratio of the three monomers. Typical properites are shown in Table 3.

Uses. The previously mentioned properties of ABS are suitable for tough consumer products (refrigerator door liners); interior automotive trim; business machine housings; telephones and other consumer electronics; luggage; and pipe, fittings, and conduit.

1.7 Polyvinyl Chloride

The catalytic polymerization of vinyl chloride yields polyvinyl chloride.[11] It is commonly referred to as PVC or vinyl and is second only to polyethylene in volume use. Normally, PVC has a low degree of crystallinity and good transparency. The high chlorine content of the polymer produces advantages in flame resistance, fair heat deflection temperature, good electrical properties, and good chemical resistance. However, the chlorine also makes PVC difficult to process. The chlorine atoms have a tendency to split out under the influence of heat during processing and heat and light during end use in finished products, producing discoloration and embrittlement. Therefore, special stabilizer systems are often used with PVC to retard degradation.

There are two major subclassifications of PVC: rigid and flexible (plasticized). In addition, there are also foamed PVC and PVC copolymers. Typical properties of PVC resins appear in Table 4.

Rigid PVC

PVC alone is a fairly good rigid polymer, but it is difficult to process and has low impact strength. Both of these properties are improved by the addition of elastomers or impact-modified graft copolymers—such as ABS and impact acrylic polymers. These improve the melt flow during processing and improve the impact strength without seriously lowering the rigidity or the heat deflection temperature.

Uses. With this improved balance of properties, rigid PVCs are used in such applications as door and window frames; pipe, fittings, and conduit; building panels and siding; rainwater gutters and down spouts; credit cards; and flooring.

Table 4 Typical Property Values for Polyvinyl Chloride Materials

Property	General Purpose	Rigid	Rigid Foam	Plasticized	Copolymer
Density (mg/m^3)	1.40	1.34–1.39	0.75	1.29–1.34	1.37
Tensile modulus (GPa)	3.45	2.41–2.45	—	—	3.15
Tensile strength (MPa)	8.7	37.2–42.4	>13.8	14–26	52–55
Elongation at break (%)	113	—	>40	250–400	—
Notched Izod (kJ/m)	0.53	0.74–1.12	>0.06	—	0.02
Heat deflection temperature at 1.81 MPa (°C)	77	73–77	65	—	65
Brittle temperature (°C)	—	—	—	−60 to −30	—
Hardness	D85 (Shore)	R107–R122 (Rockwell)	D55 (Shore)	A71–A96 (Shore)	—
Linear thermal expansion (10^{-5} mm/mm·K)	7.00	5.94	5.58	—	—
Linear mold shrinkage (in./in.)	0.003	—	—	—	—

Plasticized PVC

Flexible PVC is a plasticized material. The PVC is softened by the addition of compatible, nonvolatile, liquid plasticizers. The plasticizers, which are usually used in >20 parts per hundred resins, lower the crystallinity in PVC and act as internal lubricants to give a clear, flexible plastic. Plasticized PVC is also available in liquid formulations known as plastisols or organosols.

Uses. Plasticized PVC is used for wire and cable insulation, outdoor apparel, rainwear, flooring, interior wall covering, upholstery, automotive seat covers, garden hose, toys, clear tubing, shoes, tablecloths, and shower curtains. Plastisols are used in coating fabric, paper, and metal and rotationally cast into balls, dolls, etc.

Foamed PVC

Rigid PVC can be foamed to a low-density cellular material that is used for decorative moldings and trim. Foamed plastisols add greatly to the softness and energy absorption already inherent in plasticized PVC, giving richness and warmth to leatherlike upholstery, clothing, shoe fabrics, handbags, luggage, and auto door panels; as well as energy absorption for quiet and comfort in flooring, carpet backing, auto headliners, etc.

PVC Copolymers

Copolymerization of vinyl chloride with 10–15% vinyl acetate gives a vinyl polymer with improved flexibility and less crystallinity than PVC, making such copolymers easier to process without detracting seriously from the rigidity and heat deflection temperature. These copolymers find primary applications in flooring and solution coatings.

1.8 Poly(vinylidene chloride)

Poly(vinylidene chloride), PVDC, is prepared by the catalytic polymerization of 1,1-dichloroethylene. This crystalline polymer exhibits high strength, abrasion resistance, high melting point, better than ordinary heat resistance (100°C maximum service temperature), and outstanding impermeability to oil, grease, water vapor, oxygen, and carbon dioxide. It is used for packaging films, coatings, and monofilaments.

When the polymer is extruded into film, quenched, and oriented, the crystallinity is fine enough to produce high clarity and flexibility. These properties contribute to widespread use in packaging film, especially for food products that require impermeable barrier protection.

Poly(vinylidene chloride) and/or copolymers with vinyl chloride, alkyl acrylate, or acrylonitrile are used in coating paper, paperboard, or other films to provide more economical, impermeable materials.

A small amount of poly(vinylidene chloride) is extruded into monofilament and tape that is used in outdoor furniture upholstery.

Uses. PVDC is used in food packaging where barrier properties are needed. Applications for injection-molded grades are fittings and parts in the chemical industry. PVDC pipe is used in the disposal of waste acids.

1.9 Poly(methyl methacrylate)

The catalytic polymerization of methylmethacrylate yields poly(methyl methacrylate), PMMA, a strong, rigid, clear, amorphous polymer. PMMA has excellent resistance to weathering, low water absorption, and good electrical resistivity. PMMA properties appear in Table 5.

Uses. PMMA is used for glazing, lighting diffusers, skylights, outdoor signs, and exterior lighting lenses in cars and trucks.

1.10 Poly(ethylene terephthalate)

Poly(ethylene terephthalate), PET, is prepared from the condensation polymerization of dimethyl terephthalate and ethylene glycol. PET is a crystalline polymer that exhibits high modulus, high strength, high melting point, good electrical properties, and moisture and solvent resistance.

Uses. Primary applications of PET include blow-molded beverage bottles, fibers for wash-and-wear, wrinkle-resistant fabrics, and films that are used in food packaging, electrical applications (capacitors, etc.), magnetic recording tape, and graphic arts.

Table 5 Typical Properties of Poly(methyl methacrylate)

Property	PMMA
Density (mg/m^3)	1.18–1.19
Tensile modulus (GPa)	3.10
Tensile strength (MPa)	72
Elongation at break (%)	5
Notched Izod (kJ/m)	0.4
Heat deflection temperature at 1.81 MPa (°C)	96
Continuous service temperature (°C)	88
Hardness (Rockwell)	M90–M100
Linear thermal expansion (10^{-5} mm/mm·K)	6.3
Linear mold shrinkage (in./in.)	0.002–0.008

2 ENGINEERING THERMOPLASTICS

Engineering thermoplastics comprise a special high-performance segment of synthetic plastic materials that offer premium properties.[12,13] When properly formulated, they may be shaped into mechanically functional, semiprecision parts or structural components. Mechanically functional implies that the parts may be subjected to mechanical stress, impact, flexure, vibration, sliding friction, temperature extremes, hostile environments, etc., and continue to function.

As substitutes for metal in the construction of mechanical apparatus, engineering plastics offer advantages such as transparency, lightweight, self-lubrication, and economy in fabrication and decorating. Replacement of metals by plastic is favored as the physical properties and operating temperature ranges of plastics improve and the cost of metals and their fabrication increases.

2.1 Polyesters (Thermoplastic)

Poly(butylene terephthalate), PBT, is prepared from the condensation polymerization of butanediol with dimethyl terephthalate.[14,15] PBT is a crystalline polymer that has a fast rate of crystallization that facilitates rapid molding cycles. It seems to have a unique and favorable balance of properties between polyamides and polyacetals. PBT has low moisture absorption, extremely good self-lubricity, fatigue resistance, good solvent resistance, and good maintenance of mechanical properties at elevated temperatures. PBT resins are often used with reinforcing materials such as glass fiber to enhance strength, modulus, and heat deflection temperature. Properties appear in Table 6.

Uses. Applications of PBT include gears, rollers, bearing, housings for pumps, and appliances, impellers, pulleys, switch parts, automotive components, and electrical/electronic components. A high-density PBT is used in countertops and sinks.

2.2 Polyamides (Nylon)

The two major types of polyamides are nylon 6 and nylon 66.[16,17] Nylon 6, or polycaprolactam, is prepared by the polymerization of caprolactam. Poly(hexamethylene adipamide), or nylon 66, is derived from the condensation polymerization of hexamethylene diamine with adipic acid. Polyamides are crystalline

Table 6 Typical Properties of Poly(butylene terephthalate)

Property	PBT	PBT + 40% Glass Fiber
Density (mg/m^3)	1.300	1.600
Flexural modulus (GPa)	2.4	9.0
Flexural strength (MPa)	88	207
Elongation at break (%)	300	3
Notched Izod (kJ/m)	0.06	0.12
Heat deflection temperature at 0.45 MPa (°C)	154	232
Heat deflection temperature at 1.81 MPa (°C)	54	232
Hardness (Rockwell)	R117	M86
Linear thermal expansion (10^{-5} mm/mm·K)	9.54	1.89
Linear mold shrinkage (in./in.)	0.020	<0.007

polymers. Their key features include a high degree of solvent resistance, toughness, and fatigue resistance. Nylons do exhibit a tendency to creep under applied load. Glass fibers or mineral fillers are often used to enhance the properties of polyamides. In addition the properties of nylon are greatly effected by moisture. Properties of nylon 6 and 66 with and without glass fiber appear in Table 7.

Uses. The largest application of nylons is in fibers. Molded applications include automotive components, related machine parts (gears, cams, pulleys, rollers, boat propellers, etc.), appliance parts, and electrical insulation.

Modified Polyamides

Moisture has a profound effect on the properties of polyamides. Water acts as a plasticizer in polyamides and lowers the rigidity and strength while increasing the ductility. Moreover, this increase in moisture has a negative effect on dimensional stability. Polyamides have been modified by blending with poly(phenylene ether), PPE, in order to minimize the effect of moisture.[18–20] In PA/PPE alloys the polyamide is the continuous phase and imparts good solvent resistance. The PPE is a dispersed phase and acts as a reinforcement of the crystalline nylon matrix giving improved stiffness versus the unfilled nylon resin. Since PPE does not absorb any significant amount of moisture, the effect of moisture on properties is reduced. In addition, heat deflection temperatures have been enhanced. Properties are shown in Table 8.

Uses. PA/PPE alloys are used in automotive body panels (fenders and quarter panels); automotive wheel covers, exterior truck parts, under-the-hood automotive parts (air intake resonators, electrical junction boxes and connectors), fluid handling applications (pumps, etc.).

2.3 Polyacetals

Polyacetals are prepared via the polymerization of formaldehyde or the copolymerization of formaldehyde with ethylene oxide.[15] Polyacetals are crystalline polymers that exhibit rigidity, high strength, solvent resistance, fatigue resis-

Table 7 Typical Properties of Polyamides

Property	PA6	PA6 + 40% Glass Fiber	PA66	PA66 + 40% Glass Fiber
Density (mg/m^3)	1.130	1.460	1.140	1.440
Flexural modulus (GPa)	2.8	10.3	2.8	9.3
Flexural strength (MPa)	113	248	—	219
Elongation at break (%)	150	3	60	4
Notched Izod (kJ/m)	0.06	0.16	0.05	0.14
Heat deflection temperature at 0.45 MPa (°C)	170	218	235	260
Heat deflection temperature at 1.81 MPa (°C)	64	216	90	250
Hardness (Rockwell)	R119	M92	R121	M119
Linear thermal expansion (10^{-5} mm/mm·K)	8.28	2.16	8.10	3.42
Linear mold shrinkage (in./in.)	0.013	0.003	0.0150	0.0025

Table 8 Typical Properties of PPE/Polyamide 66 Alloys

	Unfilled		10% Glass Fiber		30% Glass Fiber	
Property	PA	PPE/PA	PA	PPE/PA	PA	PPE/PA
Density (mg/m^3)	1.14	1.10	1.204	1.163	1.37	1.33
Flexural modulus (GPa)						
Dry as molded	2.8	2.2	4.5	3.8	8.3	8.1
100% relative humidity	0.48	0.63	2.3	2.6	4.1	5.8
At 150°C	0.21	0.70	0.9	1.6	3.2	4.3
Flexural strength (MPa)						
Dry as molded	96	92	151	146	275	251
100% relative humidity	26	60	93	109	200	210
At 150°C	14	28	55	60	122	128

tance, toughness, self-lubricity, and cold-flow resistance. They also exhibit a tendency to thermally depolymerize and, hence, are difficult to flame retard. Properties are enhanced by the addition of glass fiber or mineral fillers. Typical properties appear in Table 9.

Uses. Applications of polyacetals include moving parts in appliances and machines (gears, bearings, bushings, etc.), in automobiles (door handles, etc.), plumbing and irrigation (valves, pumps, faucets, shower heads, etc.), industrial or mechanical products (rollers, bearings, gears, conveyer chains, and housings), and consumer products (cams, zippers, pen barrels, disposable lighters, and combs).

2.4 Polyphenylene Sulfide

The condensation polymerization of dichlorobenzene and sodium sulfide yields a crystalline polymer, polyphenylene sulfide (PPS).[21] It is characterized by high heat resistance, rigidity, excellent chemical resistance, low friction coefficient, good abrasion resistance, and electrical properties. PPS is somewhat difficult to process due to the very high melting temperature, relatively poor flow characteristics, and some tendency for slight cross-linking during processing. PPS resins normally contain glass fibers for mineral fillers. Properties appear in Table 10.

Table 9 Typical Properties of Polyacetals

Property	Polyacetal	Polyacetal + 40% Glass Fiber
Density (mg/m^3)	1.420	1.740
Flexural modulus (GPa)	2.7	11.0
Flexural strength (MPa	107	117
Elongation at break (%)	75	1.5
Notched Izod (kJ/m)	0.12	0.05
Heat deflection temperature at 0.45 MPa (°C)	170	167
Heat deflection temperature at 1.81 MPa (°C)	124	164
Hardness (Rockwell)	M94	R118
Linear thermal expansion (10^{-5} mm/mm·K)	10.4	3.2
Linear mold shrinkage (in./in.)	0.02	0.003

Table 10 Typical Properties of Poly(phenylene sulfide)

Property	PPS + 40% Glass Fiber
Density (mg/m^3)	1.640
Tensile modulus (GPa)	7.7
Tensile strength (MPa)	135
Elongation at break (%)	1.3
Flexural modulus (GPa)	11.7
Flexural strength (MPa)	200
Notched Izod (kJ/m)	0.08
Heat deflection temperature at 1.81 MPa (°C)	>260
Constant service temperature (°C)	232
Hardness (Rockwell)	R123
Linear thermal expansion (10^{-5} mm/mm·K)	4.0
Linear mold shrinkage (in./in.)	0.004

Uses. The reinforced materials are used in aerospace applications, chemical pump components, electrical/electronic components, appliance parts, and in automotive applications (electrical connectors, under-the-hood applications).

2.5 Polycarbonates

Most commercial polycarbonates are derived from the reaction of bisphenol A and phosgene.[22–24] Polycarbonates (PC) are transparent amorphous polymers. PCs are among the stronger, tougher, and more rigid thermoplastics. Polycarbonates also show resistance to creep and excellent electrical insulating characteristics. Polycarbonate properties are shown in Table 11.

Uses. Applications of PC include safety glazing, safety shields, nonbreakable windows, automotive taillights, electrical relay covers, various appliance parts and housings, power tool housings, automotive exterior parts, and blow-molded bottles.

Table 11 Typical Properties of Polycarbonates

Property	PC	PC + 40% Glass Fiber
Density (mg/m^3)	1.200	1.520
Tensile modulus (GPa)	2.4	11.6
Tensile strength (MPa)	65	158
Elongation at break (%)	110	4
Flexural modulus (GPa)	2.3	9.7
Flexural strength (MPa)	93	186
Notched Izod (kJ/m)	0.86	0.13
Heat deflection temperature at 0.45 MPa (°C)	138	154
Heat deflection temperature at 1.81 MPa (°C)	132	146
Constant service temperature (°C)	121	135
Hardness (Rockwell)	M70	M93
Linear thermal expansion (10^{-5} mm/mm·K)	6.74	1.67
Linear mold shrinkage (in./in.)	0.006	0.0015

Table 12 Typical Properties of Polycarbonates/ABS Blends

	PA/ABS Ratio (wt/wt)			
Properties	0/100	50/50	80/20	100/00
Density (mg/m^3)	1.06	1.13	1.17	1.20
Tensile modulus (GPa)	1.8	1.9	2.5	2.4
Tensile strength (MPa)	40	57	60	65
Elongation at break (%)	20	70	150	110
Notched Izod				
At 25°C (kJ/m)	0.30	0.69	0.75	0.86
At −20°C (kJ/m)	0.11	0.32	0.64	0.15
Heat deflection temperature				
At 1.81 MPa (°C)	80	100	113	132

Polycarbonate/ABS Alloys

PC/ABS blends are prepared by extruder blending and offer a unique balance of properties.[22] The addition of ABS improves the melt processing of the blend, which facilitate filling large, thin-walled parts. The toughness of PC—especially at low temperatures—is enhanced by the blending with ABS while maintaining the high strength and rigidity. The properties are a function of the ABS-to-polycarbonate ratio. Properties appear in Table 12.

Uses. PC/ABS is used in automotive body panels (doors) and housewares (small appliances). PC/ABS has become the resin of choice for business equipment because of the combination of processing ease and toughness.

2.6 Polysulfone

Polysulfone is prepared from the condensation polymerization of bisphenol A and dichlorodiphenyl sulfone.[25–28] The transparent, amorphous resin is characterized by excellent thermo-oxidative stability, high heat resistance, hydrolytic stability, outstanding chemical resistance (acids, bases, and alcohols), and creep resistance. Properties appear in Table 13.

Table 13 Typical Properties of Polysulfone

Property	Polysulfone
Density (mg/m^3)	1.240
Tensile modulus (GPa)	2.48
Tensile strength (MPa)	70
Elongation at break (%)	75
Flexural modulus (GPa)	2.69
Flexural strength (MPa)	106
Notched Izod (kJ/m)	0.07
Heat deflection temperature at 1.81 MPa (°C)	174
Constant service temperature (°C)	150
Hardness (Rockwell)	M69
Linear thermal expansion (10^{-5} mm/mm·K)	5.6
Linear mold shrinkage (in./in.)	0.007

Uses. Typical applications of polysulfones include microwave cookware, medical and laboratory equipment where repeated sterilization by steam is required, coffee makers, and electrical/electronic components, and chemical processing equipment.

2.7 Modified Polyphenylene Ether

Blends of poly(2,6-dimethyl phenylene ether), PPE, with styrenics (i.e., HIPS, ABS, etc.) form a family of modified PPE-based resins.[29–31] These amorphous blends cover a wide range of heat deflection temperatures, which is dependent on the PPE-to-HIPS ratio. They are characterized by outstanding dimensional stability at elevated temperatures, outstanding hydrolytic stability, long-term stability under load, and excellent dielectric properties over a wide range of frequencies and temperatures. Their properties appear in Table 14.

Uses. Applications include automotive (instrument panels, trim, etc.), TV cabinets, electrical connectors, pumps, plumbing fixtures, business machines, medical, telecommunication equipment, microwavable food packaging, and appliances.

2.8 Polyimides

Polyimides are a class of polymers prepared from the condensation reaction of a carboxylic acid anhydride with a diamine.[32] Thermoplastic and thermoset grades of polyimides are available. The thermoset polyimides are among the most heat-resistant polymers; for example, they can withstand temperatures up to 250°C. Thermoplastic polyimides, which can be processed by standard techniques, fall into two main categories—polyetherimides (PEI) and polyamide-imides (PAI).[33–35]

In general, polyimides have high heat resistance, high deflection temperatures, very good electrical properties, very good wear resistance, superior dimensional stability, outstanding flame resistance, and very high strength and rigidity. Polyimide properties appear in Table 15.

Table 14 Typical Properties of Modified Polyphenylene Ether Resins

Property	190 Grade	225 Grade	300 Grade
Density (mg/m^3)	1.080	1.090	1.060
Tensile modulus (GPa)	2.5	2.4	—
Tensile strength (MPa)	48	55	76
Elongation at break (%)	35	35	—
Flexural modulus (GPa)	2.2	2.4	2.4
Flexural strength (MPa)	57	76	104
Notched Izod (kJ/m)	0.37	0.32	0.53
Heat deflection temperature at 0.45 MPa (°C)	96	118	157
Heat deflection temperature at 1.81 MPa (°C)	88	107	149
Constant service temperature (°C)	—	95	—
Hardness (Rockwell)	R115	R116	R119
Linear thermal expansion (10^{-5} mm/mm·K)	—	—	5.9
Linear mold shrinkage (in./in.)	0.006	0.006	0.006

Table 15 Typical Properties of Polyimides

Property	Polyimide	Polyetherimide		Polyamideimide	
		Unfilled	30% Glass Fiber	Unfilled	30% Glass Fiber
Density (mg/m^3)	—	1.27	1.51	1.38	1.57
Tensile modulus (GPa)	2.65	2.97	10.3	4.83	10.7
Tensile strength (MPa)	195	97	193	117	205
Elongation at break (%)	90	60	3	10	5
Notched Izod (kJ/m)	—	0.6	0.11	0.13	0.11
Heat deflection temperature at 0.45 MPa (°C)	—	410	414	—	—
Heat deflection temperature at 1.81 MPa (°C)	—	392	410	260	274
Constant service temperature (°C)					
Hardness (Rockwell)	—	R109	M125	E78	E94
Linear thermal expansion (10^{-5} mm/mm·K)	—	5.6	2.0	3.60	1.80
Linear mold shrinkage (in./in.)	—	0.5	0.2	—	0.25

Uses. Polyimide applications include gears, bushings, bearings, seals, insulators, electrical/electronic components (printed wiring boards, connectors, etc.). PEI is used in transportation (under-the-hood temperature sensors, fuel system components, high-strength transmission and jet engine parts), medical (autoclaveable parts), electrical/electronics, packaging, appliances, industrial (heat and corrosion resistance, air and fluid handling components), cooking utensils, microwave oven components, and structural components. PAI is used in automobile transmissions (thrust washers and seal rings), parts for gas turbine engines, business machines, hot glass-handling equipment, plasma-cutting torches. Polyimide foam is used for thermal and sound-dampening insulation and seat cushions in aerospace, marine, and industrial applications.

3 FLUORINATED THERMOPLASTICS

Fluoropolymers, or fluoroplastics, are a family of fluorine-containing thermoplastics that exhibit some unusual properties.[36–37] These properties include inertness to most chemicals, resistance to high temperatures, extremely low coefficient of friction, weather resistance, and excellent dielectric properties. Mechanical properties are normally low but can be enhanced with glass or carbon fiber or molybdenum disulfide fillers. Properties are shown in Table 16.

3.1 Poly(tetrafluoroethylene)

Poly(tetrafluoroethylene), PTFE, is a crystalline, very heat resistant (up to 250°C), chemical-resistant polymer.[37] PTFE has the lowest coefficient of friction of any polymer. Poly(tetrafluoroethylene) does not soften like other thermoplastics and has to be processed by unconventional techniques (PTFE powder is compacted to the desired shape and sintered).

Uses. PTFE applications include nonstick coatings on cookware; nonlubricated bearings; chemical-resistant pipe, fitting valves, and pump parts; high-temperature electrical parts; and gaskets, seals, and packings.

Table 16 Typical Properties of Fluoropolymers

Property	PTFE	CTFE	FEP	ETFE	ECTFE
Density (mg/m^3)	2.160	2.100	2.150	1.700	1.680
Tensile modulus (GPa)	—	14.3	—	—	—
Tensile strength (MPa)	27.6	39.4	20.7	44.8	48.3
Elongation at break (%)	~275	~150	~300	100–300	200
Notched Izod (kJ/m)	—	0.27	0.15	—	—
Heat deflection temperature at 0.45 MPa (°C)	—	126	—	104	116
Heat deflection temperature at 1.81 MPa (°C)	—	75	—	71	77
Constant service temperature (°C)	260	199	204	—	150–170
Hardness	D55–65 (Shore)	D75–80 (Shore)	D55 (Shore)	D75 (Shore)	R93 (Rockwell)
Dielectric strength (MV/m)	23.6	19.7	82.7	7.9	19.3
Dielectric constant at 10^2 Hz	2.1	3.0	2.1	2.6	2.5
Dielectric constant at 10^3 Hz	2.1	2.7	—	2.6	2.5
Linear thermal expansion (10^{-5} mm/mm·K)	9.9	4.8	9.3	13.68	—
Linear mold shrinkage (in./in.)	0.033–0.053	0.008	–	–	<0.025

3.2 Poly(chlorotrifluoroethylene)

Poly(chlorotrifluoroethylene), CTFE, is less crystalline and exhibits higher rigidity and strength than PTFE.[37] Poly(chlorotrifluoroethylene) has excellent chemical resistance and heat resistance up to 200°C. Unlike PTFE, CTFE can be molded and extruded by conventional processing techniques.

Uses. CTFE applications include electrical insulation, cable jacketing, electrical and electronic coil forms, pipe and pump parts, valve diaphragms, and coatings for corrosive process equipment and other industrial parts.

3.3 Fluorinated Ethylene–Propylene

Copolymerization of tetrafluoroethylene with some hexafluoropropylene produces fluorinated ethylene–propylene polymer, FEP, which has less crystallinity, lower melting point, and improved impact strength than PTFE. FEP can be molded by normal thermoplastic techniques.[37]

Uses. FEP applications include wire insulation and jacketing, high-frequency connectors, coils, gaskets, and tube sockets.

3.4 Polyvinylidene Fluoride

Polyvinylidene fluoride, PVDF, has high tensile strength, better ability to be processed but less thermal and chemical resistance than FEP, CTFE, and PTFE.[37]

Uses. Polyvinylidene fluoride applications include seals and gaskets, diaphragms, and piping.

3.5 Poly(ethylene chlorotrifluoroethylene)

The copolymer of ethylene and chlorotrifluoroethylene is poly(ethylene chlorotrifluoroethylene), ECTFE, and has high strength, chemical and impact resistance. ECTFE can be processed by conventional techniques.

Table 17 Properties of General-Purpose Elastomers

Rubber	ASTM Nomenclature	Outstanding Characteristic	Property Deficiency	Temperature Use Range (°C)
Butadiene rubber	BR	Very flexible; resistance to wear	Sensitive to oxidation; poor resistance to fuels and oil	−100 to 90
Natural rubber	NR	Similar to BR but less resilient	Similar to BR	−50 to 80
Isoprene rubber	IR	Similar to BR but less resilient	Similar to BR	−50 to 80
Isobutylene–isoprene rubber (butyl rubber)	IIR	High flexibility; low permeability to air		−45 to 150
Chloroprene	CR	Flame resistant; fair fuel and oil resistance; increased resistance toward oxygen, ozone, heat, light	Poor low-temperature flexibility	−40 to 115
Nitrile-butadiene	NBR	Good resistance to fuels, oils, and solvents; improved abrasion resistance	Lower resilience; higher hysteresis; poor electrical properties; poorer low temperature flexibility	−45 to 80
Styrene–butadiene rubber	SBR	Relatively low cost	Less resilience; higher hysteresis; limited low temperature flexibility	−50 to 80
Ethylene-propylene copolymer	EPDM	Resistance to ozone and weathering	Poor hydrocarbon and oil resistance	−50 to <175
Polysulfide	T	Chemical resistance; resistance to ozone and weathering	Creep; low resilience	−45 to 120

Table 18 Properties of Specialty Elastomers

Elastomer	ASTM Nomenclature	Temperature Use Range (°C)	Outstanding Characteristic	Typical Applications
Silicones (polydimethylsiloxane)	MQ	−100 to 300	Wide temperature range; resistance to aging, ozone, sunlight; very high gas permeability	Seals, molded and extruded goods; adhesives, sealants; biomedical; personal care products
Fluoroelastomers	CFM	−40 to 200	Resistance to heat, oils, chemical	Seals such as O-rings, corrosion resistant coatings
Acrylic	AR	−40 to 200	Oil, oxygen, ozone, and sunlight resistance	Seals, hose
Epichlorohydrin	ECO	−18 to 150	Resistance to oil, fuels; some flame resistance; low gas permeability	Hose, tubing, coated fabrics, vibration isolators
Chlorosulfonated	CSM	−40 to 150	Resistance to oil, ozone weathering, oxidizing chemicals	Automotive hose, wire and cable, linings for reservoirs
Chlorinated polyethylene	CM	−40 to 150	Resistance to oils, ozone, chemicals	Impact modifier, automotive applications
Ethylene–acrylic		−40 to 175	Resistance to ozone, weathering	Seals, insulation, vibration damping
Propylene oxide		−60 to 150	Low temperature properties	Motor mounts

Uses. Poly(ethylene chlorotrifluoroethylene) applications included wire and cable coatings, chemical-resistant coatings and linings, molded lab ware, and medical packing.

3.6 Poly(vinyl fluoride)

Poly(vinyl fluoride) films exhibit excellent outdoor durability. It is the least chemical-resistant fluoropolymer.[37]

Uses. Poly(vinyl fluoride) uses include glazing, lighting, and coatings on presurfaced exterior building panels.

4 THERMOSETS

Thermosetting resins are used in molded and laminated plastics.[38] They are first polymerized into a low-molecular-weight linear or slightly branched polymer or oligomer, which is still soluble, fusible, and highly reactive during final processing. Thermoset resins are generally highly filled with mineral fillers and glass fibers. Thermosets are generally catalyzed and/or heated to finish the polymerization reaction, cross-linking them to almost infinite molecular weight. This step is often referred to as cure. Such cured polymers cannot be reprocessed or reshaped.

The high filler loading and the high cross-link density of thermoset resins results in very high densities and very low ductility, but very high rigidity and good chemical resistance.

4.1 Phenolic Resins

Phenolic resins combine the high reactivity of phenol and formaldehyde to form prepolymers and oligomers called resoles and novolacs. These materials are combined with fibrous fillers to give a phenolic resin, which when heated provides rapid, complete cross-linking into highly cured structures. The high cross-linked aromatic structure has high hardness, rigidity, strength, heat resistance, chemical resistance, and good electrical properties.

Uses. Phenolic applications include automotive uses (distributor caps, rotors, brake linings), appliance parts (pot handles, knobs, bases, electrical/electronic components (connectors, circuit breakers, switches), and as an adhesive in laminated materials (e.g., plywood).

4.2 Epoxy Resins

The most common epoxy resins are prepared from the reaction of bisphenol A and epichlorohydrin to yield low-molecular-weight resins that are liquid either at room temperature or on warming. Each polymer chain usually contains two or more epoxide groups. The high reactivity of the epoxide groups with amines, anhydrides, and other curing agents provides facile conversion into highly cross-linked materials. Cured epoxy resins exhibit hardness, strength, heat resistance, electrical resistance, and broad chemical resistance.

Uses. Epoxy resins are used in glass reinforced, high-strength composites in aerospace, pipes, tanks, pressure vessels; encapsulation or casting of various electrical and electronic components (printed wiring boards, etc.); adhesives;

protective coatings in appliances, flooring, and industrial equipment; and sealants.

4.3 Unsaturated Polyesters

Unsaturated polyesters are prepared by the condensation polymerization of various diols and maleic anhydride to give a very viscous liquid that is dissolved in styrene monomer. The addition of styrene lowers the viscosity to a level suitable for impregnation and lamination of glass fibers. The low-molecular-weight polyester has numerous fumarate ester units that provide easy reactivity with styrene monomer. Properly formulated glass-reinforced unsaturated polyesters are commonly referred to as sheet molding compound (SMC), or reinforced plastics. In combination with reinforcing materials such as glass fiber, cured resins offer outstanding strength, high rigidity, impact resistance, high strength-to-weight ratio, and chemical resistance. SMC typically is formulated with 50% calcium carbonate filler, 25% long glass fiber, and 25% unsaturated polyester. The highly filled nature of SMC results in high density and brittle easily pitted surface.

Bulk molding compound (BMC) is formulated similar to SMC except $\frac{1}{4}$-in. chopped glass is used. The shorter glass length gives easier process but lower strength and impact.

Uses. The prime use of unsaturated polyesters is in combination with glass fibers in high-strength composites and in SMC and BMC materials. The applications include transportation markets (large body parts for automobiles, trucks, trailers, buses, and aircraft), marine markets (small- to medium-sized boat hulls and associated marine equipment), building panels, housing and bathroom components (bathtub and shower stalls), appliances, and electrical/electronic components.

4.4 Alkyd Resins

Alkyd resins are based on branched prepolymers from glycerol, phthalic anhydride, and glyceryl esters of fatty acids. Alkyds have excellent heat resistance, are dimensionally stable at high temperatures, and have excellent dielectric strength (>14 MV/m), high resistance to electrical leakage, and excellent arc resistance.

Uses. Alkyd resin applications include drying oils in enamel paints, lacquers for automobiles and appliances; and molding compounds when formulated with reinforcing fillers for electrical applications (circuit breaker insulation, encapsulation of capacitors and resistors, and coil forms).

4.5 Diallyl Phthalate

Diallyl phthalate (DAP) is the most widely used compound in the allylic family. The neat resin is a medium-viscosity liquid. These low-molecular-weight prepolymers can be reinforced and compression molded into highly cross-linked, completely cured products.

The most outstanding properties of DAP are excellent dimensional stability and high insulation resistance. In addition, DAP has high dielectric strength, excellent arc resistance, and chemical resistance.

Uses. DAP applications include electronic parts, electrical connectors, bases and housings. DAP is also used as a coating and impregnating material.

4.6 Amino Resins

The two main members of the amino family of thermosets are the melamine and urea resins. They are prepared from the reaction of melamine and urea with formaldehyde. In general, these materials exhibit extreme hardness, scratch resistance, electrical resistance, and chemical resistance.

Uses. Melamine resins find use in colorful, rugged dinnerware, decorative laminates (countertops, tabletops, and furniture surfacing), electrical applications (switchboard panels, circuit breaker parts, arc barriers, and armature and slot wedges), and adhesives and coatings.

Urea resins are used in particleboard binders, decorative housings, closures, elecrical parts, coatings, and paper and textile treatment.

5 GENERAL-PURPOSE ELASTOMERS

Elastomers are polymers that can be stretched substantially beyond their original length and can retract rapidly and forcibly to essentially their original dimensions (on release of the force).[39,40]

The optimum properties and/or economics of many rubbers are obtained through formulating with reinforcing agents, fillers, extending oils, vulcanizing agents, antioxidants, pigments, etc. End-use markets for formulated rubbers include automotive tire products (including tubes, retread applications, valve stems, and inner liners), adhesives, cements, caulks, sealants, latex foam products, hose (automotive, industrial, and consumer applications), belting (V-conveyor and trimming), footwear (heels, soles, slab stock, boots, and canvas), molded, extruded, and calendered products (athletic goods, flooring, gaskets, household products, O-rings, blown sponge, thread, and rubber sundries). A list of general-purpose elastomers and properties is summarized in Table 17.

6 SPECIALTY ELASTOMERS

Specialty rubbers offer higher performance over general-purpose rubbers and find use in more demanding applications.[41] They are more costly and hence are produced in smaller volumes. Properties and uses are summarized in Table 18.

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CHAPTER 12

COMPOSITE MATERIALS

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1 INTRODUCTION

The development of composite materials and related design and manufacturing technologies is one of the most important advances in the history of materials. Composites are multifunctional materials having unprecedented mechanical and physical properties that can be tailored to meet the requirements of a particular application. Many composites also exhibit great resistance to high-temperature corrosion and oxidation and wear. These unique characteristics provide the mechanical engineer with design opportunities not possible with conventional monolithic (unreinforced) materials. Composites technology also makes possible the use of an entire class of solid materials, ceramics, in applications for which monolithic versions are unsuited because of their great strength scatter and poor resistance to mechanical and thermal shock. Further, many manufacturing processes for composites are well adapted to the fabrication of large, complex structures, which allows consolidation of parts, reducing manufacturing costs.

Composites are important materials that are now used widely, not only in the aerospace industry, but also in a large and increasing number of commercial mechanical engineering applications, such as internal combustion engines; machine components; thermal control and electronic packaging; automobile, train, and aircraft structures and mechanical components, such as brakes, drive shafts, flywheels, tanks, and pressure vessels; dimensionally stable components; process industries equipment requiring resistance to high-temperature corrosion, oxida-

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tion, and wear; offshore and onshore oil exploration and production; marine structures; sports and leisure equipment; and biomedical devices.

It should be noted that biological structural materials occurring in nature are typically some type of composite. Common examples are wood, bamboo, bone, teeth, and shell. Further, use of artificial composite materials is not new. Straw-reinforced mud bricks were employed in biblical times. Using modern terminology, discussed later, this material would be classified as an organic fiber-reinforced ceramic matrix composite.

In this chapter, we consider the properties of reinforcements and matrix materials (Section 2) and the properties of composites (Section 3).

1.1 Classes and Characteristics of Composite Materials

There is no universally accepted definition of a composite material. For the purpose of this work, we consider a composite to be a material consisting of two or more distinct phases, bonded together.[1]

Solid materials can be divided into four categories: polymers, metals, ceramics, and carbon, which we consider as a separate class because of its unique characteristics. We find both reinforcements and matrix materials in all four categories. This gives us the ability to create a limitless number of new material systems with unique properties that cannot be obtained with any single monolithic material. Table 1 shows the types of material combinations now in use.

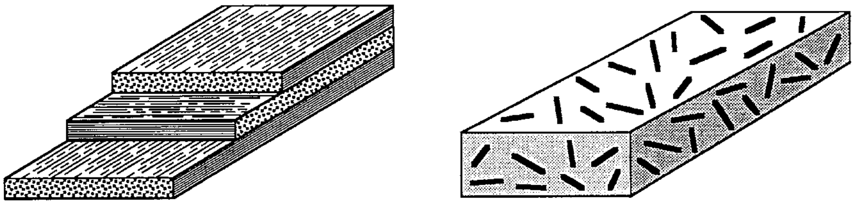
Composites are usually classified by the type of material used for the matrix. The four primary categories of composites are polymer matrix composites (PMCs), metal matrix composites (MMCs), ceramic matrix composites (CMCs), and carbon/carbon composites (CCCs). At this time, PMCs are the most widely used class of composites. However, there are important applications of the other types, which are indicative of their great potential in mechanical engineering applications.

Figure 1 shows the main types of reinforcements used in composite materials: aligned continuous fibers, discontinuous fibers, whiskers (elongated single crystals), particles, and numerous forms of fibrous architectures produced by textile technology, such as fabrics and braids. Increasingly, designers are using hybrid composites that combine different types of reinforcements to achieve more efficiency and to reduce cost.

A common way to represent fiber-reinforced composites is to show the fiber and matrix separated by a slash. For example, carbon fiber-reinforced epoxy is typically written "carbon/epoxy," or, "C/Ep." We represent particle reinforcements by enclosing them in parentheses followed by "p"; thus, silicon carbide (SiC) particle-reinforced aluminum appears as "(SiC)p/Al."

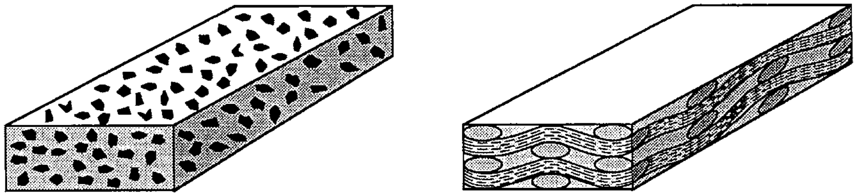
Table 1 Types of Composite Materials

	Matrix			
Reinforcement	Polymer	Metal	Ceramic	Carbon
Polymer	X	X	X	X
Metal	X	X	X	X
Ceramic	X	X	X	X
Carbon	X	X	X	X



Continuous Fibers

Discontinuous Fibers, Whiskers



Particles

Fabric, Braid, Etc.

Fig. 1 Reinforcement forms.

Composites are strongly heterogeneous materials; that is, the properties of a composite vary considerably from point to point in the material, depending on which material phase the point is located in. Monolithic ceramics and metallic alloys are usually considered to be homogeneous materials, to a first approximation.

Many artificial composites, especially those reinforced with fibers, are anisotropic, which means their properties vary with direction (the properties of isotropic materials are the same in every direction). This is a characteristic they share with a widely used natural fibrous composite, wood. As for wood, when structures made from artificial fibrous composites are required to carry load in more than one direction, they are used in laminated form.

Many fiber-reinforced composites, especially PMCs, MMCs, and CCCs, do not display plastic behavior as metals do, which makes them more sensitive to stress concentrations. However, the absence of plastic deformation does not mean that composites are brittle materials like monolithic ceramics. The heterogeneous nature of composites results in complex failure mechanisms that impart toughness. Fiber-reinforced materials have been found to produce durable, reliable structural components in countless applications. The unique characteristics of composite materials, especially anisotropy, require the use of special design methods.

1.2 Comparative Properties of Composite Materials

There are a large and increasing number of materials that fall in each of the four types of composites, making generalization difficult. However, as a class of materials, composites tend to have the following characteristics: high strength; high modulus; low density; excellent resistance to fatigue, creep, creep rupture, corrosion, and wear; and low coefficient of thermal expansion (CTE). As for monolithic materials, each of the four classes of composites has its own particular attributes. For example, CMCs tend to have particularly good resistance to corrosion, oxidation, and wear, along with high-temperature capability.

For applications in which both mechanical properties and low weight are important, useful figures of merit are specific strength (strength divided by specific gravity or density) and specific stiffness (stiffness divided by specific gravity or density). Figure 2 presents specific stiffness and specific tensile strength of conventional structural metals (steel, titanium, aluminum, magnesium, and beryllium), two engineering ceramics (silicon nitride and alumina), and selected composite materials. The composites are PMCs reinforced with selected contin-

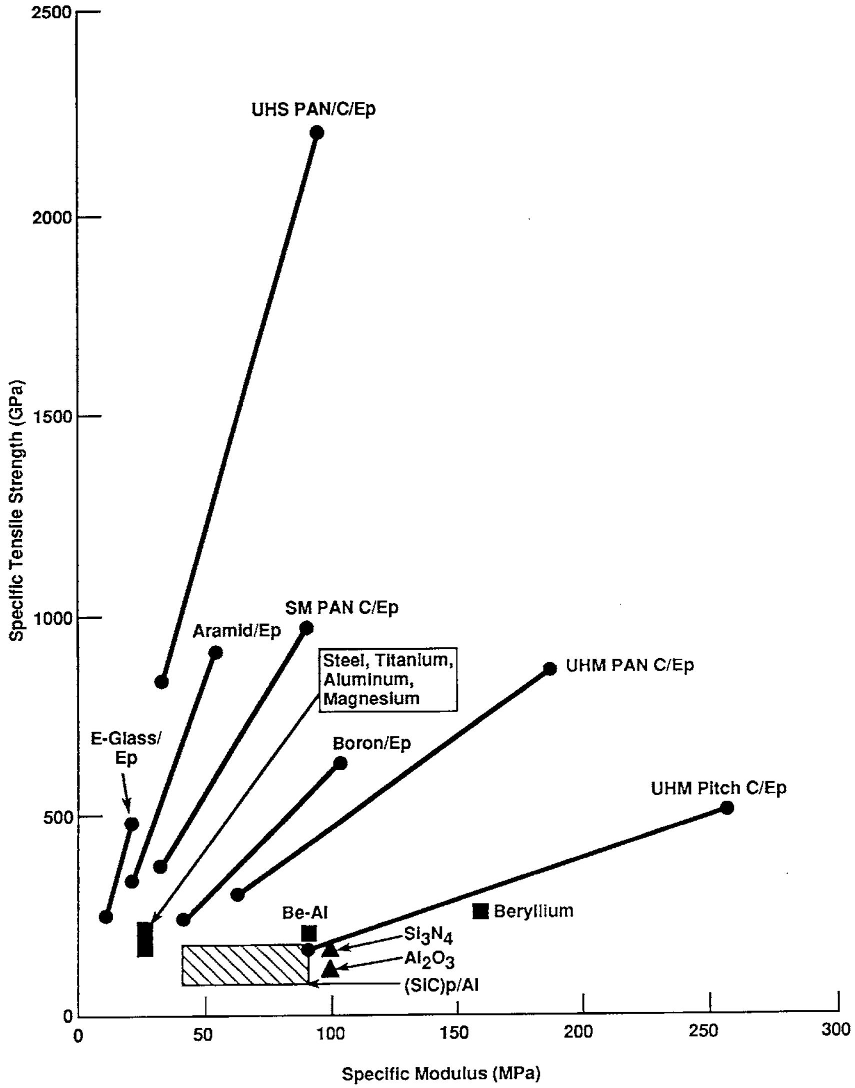


Fig. 2 Specific tensile strength (tensile strength divided by density) as a function of specific modulus (modulus divided by density) of composite materials and monolithic metals and ceramics.

uous fibers—carbon, aramid, E-glass, and boron—and an MMC, aluminum containing silicon carbide particles. Also shown is beryllium–aluminum, which can be considered a type of metal matrix composite, rather than an alloy, because the mutual solubility of the constituents at room temperature is low.

The carbon fibers represented in Figure 2 are made from several types of precursor materials: polyacrilonitrile (PAN), petroleum pitch, and coal tar pitch. Characteristics of the two types of pitch-based fibers tend to be similar but very different from those made from PAN. Several types of carbon fibers are represented: standard-modulus (SM) PAN, ultrahigh-strength (UHS) PAN, ultrahigh-modulus (UHM) PAN, and ultrahigh-modulus (UHM) pitch. These fibers are discussed in Section 2. It should be noted that there are dozens of different kinds of commercial carbon fibers, and new ones are continually being developed.

Because the properties of fiber-reinforced composites depend strongly on fiber orientation, fiber-reinforced polymers are represented by lines. The upper end corresponds to the axial properties of a unidirectional laminate, in which all the fibers are aligned in one direction. The lower end represents a quasi-isotropic laminate having equal stiffness and approximately equal strength characteristics in all directions in the plane of the fibers.

As Figure 2 shows, composites offer order-of-magnitude improvements over metals in both specific strength and stiffness. It has been observed that order-of-magnitude improvements in key properties typically produce revolutionary effects in a technology. Consequently, it is not surprising that composites are having such a dramatic influence in engineering applications.

In addition to their exceptional static strength properties, fiber-reinforced polymers also have excellent resistance to fatigue loading. Figure 3 shows how the number of cycles to failure (*N*) varies with maximum stress (*S*) for aluminum and selected unidirectional PMCs subjected to tension–tension fatigue. The ratio of minimum stress to maximum stress (*R*) is 0.1. The composites consist of epoxy matrices reinforced with key fibers: aramid, boron, SM carbon, high-

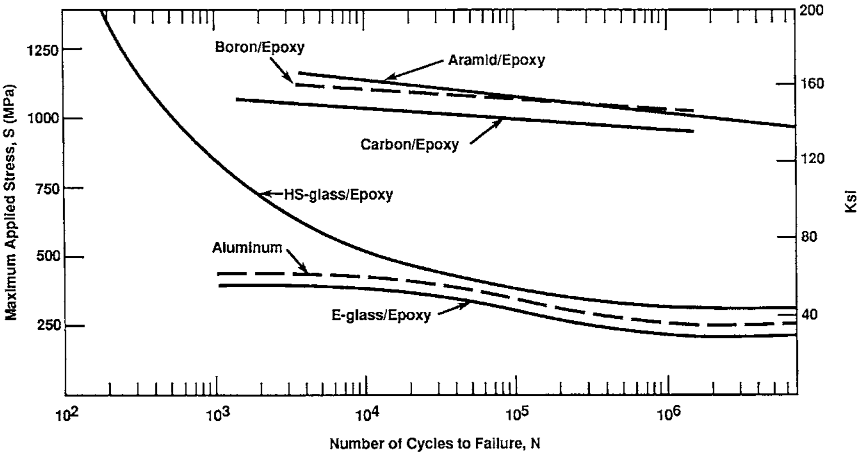


Fig. 3 Number of cycles to failure as a function of maximum stress for aluminum and unidirectional polymer matrix composites subjected to tension–tension fatigue with a stress ratio, $R = 0.1$. (From Ref. 2. Copyright ASTM. Reprinted with permission.)

strength (HS) glass, and E-glass. Because of their excellent fatigue resistance, composites have largely replaced metals in fatigue-critical aerospace applications, such as helicopter rotor blades. Composites also are being used in commercial fatigue-critical applications, such as automobile springs.

The outstanding mechanical properties of composite materials have been a key reason for their extensive use in structures. However, composites also have important physical properties, especially low, tailorable CTE and high-thermal conductivity, that are key reasons for their selection in an increasing number of applications.

Many composites, such as PMCs reinforced with carbon and aramid fibers, and silicon carbide particle-reinforced aluminum, have low CTEs, which are advantageous in applications requiring dimensional stability. By appropriate selection of reinforcements and matrix materials, it is possible to produce composites with near-zero CTEs.

Coefficient of thermal expansion tailorability provides a way to minimize thermal stresses and distortions that often arise when dissimilar materials are joined. For example, Fig. 4 shows how the CTE of silicon carbide particle-reinforced aluminum varies with particle content. By varying the amount of reinforcement, it is possible to match the CTEs of a variety of key engineering materials, such as steel, titanium, and alumina (aluminum oxide).

The ability to tailor CTE is particularly important in applications such as electronic packaging, where thermal stresses can cause failure of ceramic substrates, semiconductors, and solder joints.

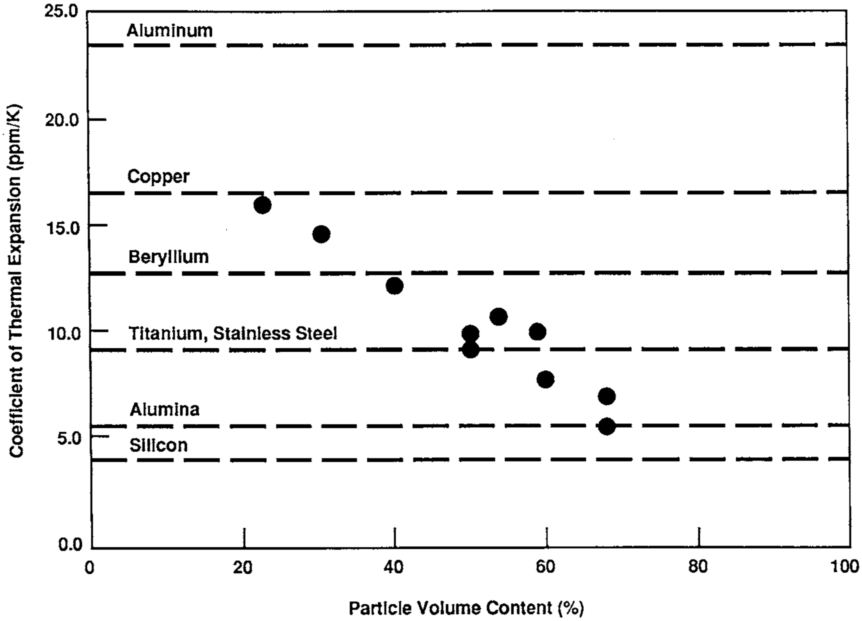


Fig. 4 Variation of coefficient of thermal expansion with particle volume fraction for silicon carbide particle-reinforced aluminum. (From Ref. 3, page 17. Copyright 1992 by TMS.)

Another unique and increasingly important property of some composites is their exceptionally high thermal conductivity. This is leading to increasing use of composites in applications for which heat dissipation is a key design consideration. In addition, the low densities of composites make them particularly advantageous in thermal control applications for which weight is important, such as laptop computers, avionics, and spacecraft components, such as radiators.

There are a large and increasing number of thermally conductive composites, which are discussed in Section 3. One of the most important types of reinforcements for these materials is pitch fibers. Figure 5 shows how thermal conductivity varies with electrical resistivity for conventional metals and carbon fibers. It can be seen that PAN-based fibers have relatively low thermal conductivities. However, pitch-based fibers with thermal conductivities more than twice that of copper are commercially available. These reinforcements also have very high stiffnesses and low densities. At the upper end of the carbon fiber curve are fibers made by chemical vapor deposition (CVD). Fibers made from another form of carbon, diamond, also have the potential for thermal conductivities in the range of 2000 W/m · K (1160 Btu/h · ft · °F).

1.3 Manufacturing Considerations

Composites also offer a number of significant manufacturing advantages over monolithic metals and ceramics. For example, fiber-reinforced polymers and ceramics can be fabricated in large, complex shapes that would be difficult or impossible to make with other materials. The ability to fabricate complex shapes allows consolidation of parts, which reduces machining and assembly costs. Some processes allow fabrication of parts to their final shape (net shape) or close to their final shape (near-net shape), which also produces manufacturing

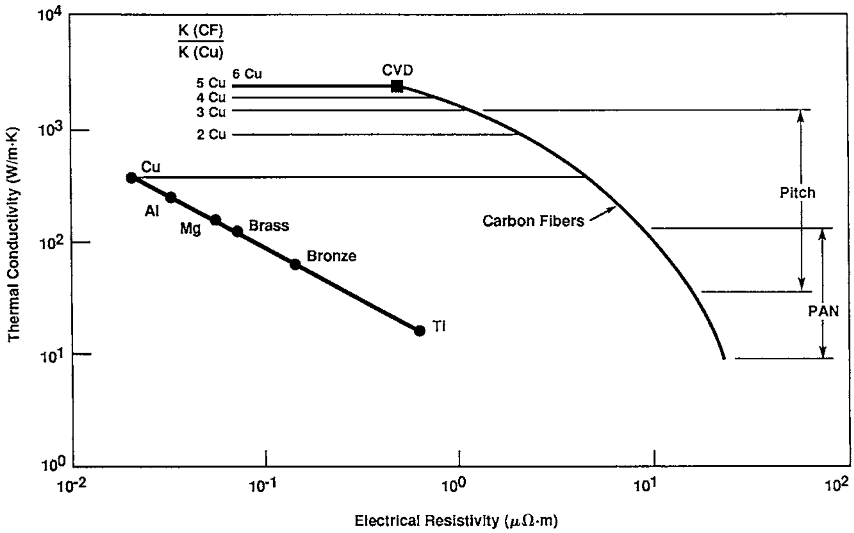


Fig. 5 Thermal conductivity as a function of electrical resistivity of metals and carbon fibers (adapted from one of Amoco Performance Products).

cost savings. The relative ease with which smooth shapes can be made is a significant factor in the use of composites in aircraft and other applications for which aerodynamic considerations are important.

2 REINFORCEMENTS AND MATRIX MATERIALS

As discussed in Section 1, we divide solid materials into four classes: polymers, metals, ceramics, and carbon. There are reinforcements and matrix materials in each category. In this section, we consider the characteristics of key reinforcements and matrices.

There are important issues that must be discussed before we present constituent properties. The conventional materials used in mechanical engineering applications are primarily structural metals, for most of which there are industry and government specifications. The situation is very different for composites. Most reinforcements and matrices are proprietary materials for which there are no industry standards. This is similar to the current status of ceramics. The situation is further complicated by the fact that there are many test methods in use to measure mechanical and physical properties of reinforcements and matrix materials. As a result, there are often conflicting material property data in the usual sources, published papers, and manufacturers' literature. The data presented in this chapter represent a carefully evaluated distillation of information from many sources. The principal sources are listed in the bibliography and references. In view of the uncertainties discussed, the properties presented in this section should be considered approximate values.

Because of the large number of matrix materials and reinforcements, we are forced to be selective. Further, space limitations prevent presentation of a complete set of properties. Consequently, properties cited are room temperature values, unless otherwise stated.

2.1 Reinforcements

The four key types of reinforcements used in composites are continuous fibers, discontinuous fibers, whiskers (elongated single crystals), and particles (Fig. 1). Continuous, aligned fibers are the most efficient reinforcement form and are widely used, especially in high-performance applications. However, for ease of fabrication and to achieve specific properties, such as improved through-thickness strength, continuous fibers are converted into a wide variety of reinforcement forms using textile technology. Key among them at this time are two-dimensional and three-dimensional fabrics and braids.

Fibers

The development of fibers with unprecedented properties has been largely responsible for the great importance of composites and the revolutionary improvements in properties compared to conventional materials that they offer. The key fibers for mechanical engineering applications are glasses, carbons (also called graphites), several types of ceramics, and high-modulus organics. Most fibers are produced in the form of multifilament bundles called strands or ends in their untwisted forms, and yarns when twisted. Some fibers are produced as monofilaments, which generally have much larger diameters than strand filaments.

Table 2 presents properties of key fibers, which are discussed in the following subsections.

Fiber strength requires some discussion. Most of the key fibrous reinforcements are made of brittle ceramics or carbon. It is well known that the strengths of monolithic ceramics decrease with increasing material volume because of the increasing probability of finding strength-limiting flaws. This is called size effect. As a result of size effect, fiber strength typically decreases monotonically with increasing gauge length (and diameter). Flaw sensitivity also results in considerable strength scatter at a fixed test length. Consequently, there is no single value that characterizes fiber strength. This is also true of key organic reinforcements, such as aramid fibers. Consequently, the values presented in Table 2 should be considered approximate values and are useful primarily for comparative purposes. Note that, because unsupported fibers buckle under very low stresses, it is very difficult to measure their inherent compression strength, and these properties are almost never reported. Instead, composite compression strength is measured directly.

Glass Fibers. Glass fibers are used primarily to reinforce polymers. The leading types of glass fibers for mechanical engineering applications are E-glass and high-strength (HS) glass. E-glass fibers, the first major composite reinforcement, were originally developed for electrical insulation applications (that is the origin of the "E"). E-glass is, by many orders of magnitude, the most widely used of all fibrous reinforcements. The primary reasons for this are its low cost and early development compared to other fibers. Glass fibers are produced as multifilament bundles. Filament diameters range from 3–20 μm (118–787 μin.). Table 2 presents representative properties of E-glass and HS glass fibers.

Table 2 Properties of Key Reinforcing Fibers

Fiber	Density [g/cm^3 (Pci)]	Axial Modulus [GPa (Msi)]	Tensile Strength [MPa (ksi)]	Axial Coefficient of Thermal Expansion [ppm/K (ppm/°F)]	Axial Thermal Conductivity [W/m · K]
E-glass	2.6 (0.094)	70 (10)	2000 (300)	5 (2.8)	0.9
HS glass	2.5 (0.090)	83 (12)	4200 (650)	4.1 (2.3)	0.9
Aramid	1.4 (0.052)	124 (18)	3200 (500)	−5.2 (−2.9)	0.04
Boron	2.6 (0.094)	400 (58)	3600 (520)	4.5 (2.5)	—
SM carbon (PAN)	1.7 (0.061)	235 (34)	3200 (500)	−0.5 (−0.3)	9
UHM carbon (PAN)	1.9 (0.069)	590 (86)	3800 (550)	−1 (−0.6)	18
UHS carbon (PAN)	1.8 (0.065)	290 (42)	7000 (1000)	−1.5 (−0.8)	160
UHM carbon (pitch)	2.2 (0.079)	895 (130)	2200 (320)	−1.6 (−0.9)	640
UHK carbon (pitch)	2.2 (0.079)	830 (120)	2200 (320)	−1.6 (−0.9)	1100
SiC monofilament	3.0 (0.11)	400 (58)	3600 (520)	4.9 (2.7)	—
SiC multifilament	3.0 (0.11)	400 (58)	3100 (450)	—	—
Si–C–O	2.6 (0.094)	190 (28)	2900 (430)	3.9 (2.2)	1.4
Si–Ti–C–O	2.4 (0.087)	190 (27)	3300 (470)	3.1 (1.7)	—
Aluminum oxide	3.9 (0.14)	370 (54)	1900 (280)	7.9 (4.4)	—
High-density polyethylene	0.97 (0.035)	172 (25)	3000 (440)	—	—

E-glass fibers have relatively low elastic moduli compared to other reinforcements. In addition, E-glass fibers are susceptible to creep and creep (stress) rupture. HS glass is stiffer and stronger than E-glass and has better resistance to fatigue and creep.

The thermal and electrical conductivities of glass fibers are low, and glass fiber-reinforced PMCs are often used as thermal and electrical insulators. The CTE of glass fibers is also low compared to most metals.

Carbon (Graphite) Fibers. Carbon fibers, commonly called graphite fibers in the United States, are used as reinforcements for polymers, metals, ceramics, and carbon. There are dozens of commercial carbon fibers, with a wide range of strengths and moduli. As a class of reinforcements, carbon fibers are characterized by high stiffness and strength, and low density and CTE. Fibers with tensile moduli as high as 895 GPa (130 Msi) and with tensile strengths of 7000 MPa (1000 ksi) are commercially available. Carbon fibers have excellent resistance to creep, stress rupture, fatigue, and corrosive environments, although they oxidize at high temperatures. Some carbon fibers also have extremely high thermal conductivities—many times that of copper. This characteristic is of considerable interest in electronic packaging and other applications where thermal control is important. Carbon fibers are the workhorse reinforcements in high-performance aerospace and commercial PMCs and some CMCs. Of course, as the name suggests, carbon fibers are also the reinforcements in carbon/carbon composites.

Most carbon fibers are highly anisotropic. Axial stiffness, tension and compression strength, and thermal conductivity are typically much greater than the corresponding properties in the radial direction. Carbon fibers generally have small, negative axial CTEs (which means that they get shorter when heated) and positive radial CTEs. Diameters of common reinforcing fibers, which are produced in the form of multifilament bundles, range from 4–10 μm (160–390 μin.). Carbon fiber stress–strain curves tend to be nonlinear. Modulus increases under increasing tensile stress and decreases under increasing compressive stress.

Carbon fibers are made primarily from three key precursor materials: polyacrylonitrile (PAN), petroleum pitch, and coal tar pitch. Rayon-based fibers, once the primary CCC reinforcement, are far less common in new applications. Experimental fibers also have been made by chemical vapor deposition. Some of these have reported axial thermal conductivities as high as 2000 W/m · K, five times that of copper.

PAN-based materials are the most widely used carbon fibers. There are dozens on the market. Fiber axial moduli range from 235 GPa (34 Msi) to 590 GPa (85 Msi). They generally provide composites with excellent tensile and compressive strength properties, although compressive strength tends to drop off as modulus increases. Fibers having tensile strengths as high as 7 GPa (1 Msi) are available. Table 2 presents properties of three types of PAN-based carbon fibers and two types of pitch-based carbon fibers. The PAN-based fibers are standard modulus (SM), ultrahigh strength (UHS) and ultrahigh modulus (UHM). SM PAN fibers are the most widely used type of carbon fiber reinforcement. They are one of the first types commercialized and tend to be the least expensive. UHS PAN carbon fibers are the strongest type of another widely used class of carbon fiber, usually called intermediate modulus (IM) because the axial modulus of these fibers falls between those of SM and modulus carbon fibers.

A key advantage of pitch-based fibers is that they can be produced with much higher axial moduli than those made from PAN precursors. For example, UHM pitch fibers with moduli as high as 895 GPa (130 Msi) are available. In addition, some pitch fibers, which we designate UHK, have extremely high axial thermal conductivities. There are commercial UHK fibers with a nominal axial thermal conductivity of 1100 W/m · K, almost three times that of copper. However, composites made from pitch-based carbon fibers generally are somewhat weaker in tension and shear, and much weaker in compression, than those using PAN-based reinforcements.

Boron Fibers. Boron fibers are primarily used to reinforce polymers and metals. Boron fibers are produced as monofilaments (single filaments) by chemical vapor deposition of boron on a tungsten wire or carbon filament, the former being the most widely used. They have relatively large diameters, 100–140 μm (4000–5600 μin.), compared to most other reinforcements. Table 2 presents representative properties of boron fibers having a tungsten core and diameter of 140 μm. The properties of boron fibers are influenced by the ratio of overall fiber diameter to that of the tungsten core. For example, fiber specific gravity is 2.57 for 100-μm fibers and 2.49 for 140-μm fibers.

Fibers Based on Silicon Carbide. Silicon-carbide-based fibers are primarily used to reinforce metals and ceramics. There are a number of commercial fibers based on silicon carbide. One type, a monofilament, is produced by chemical vapor deposition of high-purity silicon carbide on a carbon monofilament core. Some versions use a carbon-rich surface layer that serves as a reaction barrier. There are a number of multifilament silicon-carbide-based fibers made by pyrolysis of polymers. Some of these contain varying amounts of silicon, carbon and oxygen, titanium, nitrogen, zirconium, and hydrogen. Table 2 presents properties of selected silicon-carbide-based fibers.

Fibers Based on Alumina. Alumina-based fibers are primarily used to reinforce metals and ceramics. Like silicon–carbide-based fibers, they have a number of different chemical formulations. The primary constituents, in addition to alumina, are boria, silica, and zirconia. Table 2 presents properties of high-purity alumina fibers.

Aramid Fibers. Aramid, or aromatic, polyamide fibers are high-modulus organic reinforcements primarily used to reinforce polymers and for ballistic protection. There are a number of commercial aramid fibers produced by several manufacturers. Like other reinforcements, they are proprietary materials with different properties. Table 2 presents properties of one of the most widely used aramid fibers.

High-Density Polyethylene Fibers. High-density polyethylene fibers are primarily used to reinforce polymers and for ballistic protection. Table 2 presents properties of a common reinforcing fiber. The properties of high-density polyethylene tend to decrease significantly with increasing temperature, and they tend to creep significantly under load, even at low temperatures.

2.2 Matrix Materials

The four classes of matrix materials are polymers, metals, ceramics, and carbon. Table 3 presents representative properties of selected matrix materials in each category. As the table shows, the properties of the four types differ substantially. These differences have profound effects on the properties of the composites using them. In this section, we examine characteristics of key materials in each class.

Polymer Matrix Materials

There are two major classes of polymers used as matrix materials: thermosets and thermoplastics. Thermosets are materials that undergo a curing process during part fabrication, after which they are rigid and cannot be reformed. Thermoplastics, on the other hand, can be repeatedly softened and reformed by application of heat. Thermoplastics are often subdivided into several types: amorphous, crystalline, and liquid crystal. There are numerous types of polymers in both classes. Thermosets tend to be more resistant to solvents and corrosive environments than thermoplastics, but there are exceptions to this rule. Resin selection is based on design requirements, as well as manufacturing and cost considerations. Table 4 presents representative properties of common matrix polymers.

Polymer matrices generally are relatively weak, low-stiffness, viscoelastic materials. The strength and stiffness of PMCs come primarily from the fiber phase. One of the key issues in matrix selection is maximum service temperature. The properties of polymers decrease with increasing temperature. A widely used measure of comparative temperature resistance of polymers is glass transition temperature (T_g), which is the approximate temperature at which a polymer transitions from a relatively rigid material to a rubbery one. Polymers typically suffer significant losses in both strength and stiffness above their glass transition temperatures. New polymers with increasing temperature capability are continually being developed, allowing them to compete with a wider range of metals. For example, carbon fiber-reinforced polyimides have replaced titanium in some aircraft gas turbine engine parts.

An important consideration in selection of polymer matrices is their moisture sensitivity. Resins tend to absorb water, which causes dimensional changes and reduction of elevated temperature strength and stiffness. The amount of moisture absorption, typically measured as percent weight gain, depends on the polymer and relative humidity. Resins also desorb moisture when placed in a drier atmosphere. The rate of absorption and desorption depends strongly on temperature. The moisture sensitivity of resins varies widely; some are very resistant.

In a vacuum, resins outgas water and organic and inorganic chemicals, which can condense on surfaces with which they come in contact. This can be a problem in optical systems and can affect surface properties critical for thermal control, such as absorptivity and emissivity. Outgassing can be controlled by resin selection and baking out the component.

Thermosetting Resins. The key types of thermosetting resins used in composites are epoxies, bismaleimides, thermosetting polyimides, cyanate esters, thermosetting polyesters, vinyl esters, and phenolics.

Table 3 Properties of Selected Matrix Materials

Material	Class	Density [g/cm^3 (Pci)]	Modulus [GPa (Msi)]	Tensile Strength [MPa (ksi)]	Tensile Failure Strain (%)	Thermal Conductivity [W/m · K (BTU/h · ft · °F)]	Coefficient of Thermal Expansion [ppm/K (ppm/°F)]
Epoxy	Polymer	1.8 (0.065)	3.5 (0.5)	70 (10)	3	0.1 (0.06)	60 (33)
Aluminum (6061)	Metal	2.7 (0.098)	69 (10)	300 (43)	10	180 (104)	23 (13)
Titanium (6Al–4V)	Metal	4.4 (0.16)	105 (15.2)	1100 (160)	10	16 (9.5)	9.5 (5.3)
Silicon carbide	Ceramic	2.9 (0.106)	520 (75)	—	<0.1	81 (47)	4.9 (2.7)
Alumina	Ceramic	3.9 (0.141)	380 (55)	—	<0.1	20 (120)	6.7 (3.7)
Glass (borosilicate)	Ceramic	2.2 (0.079)	63 (9)	—	<0.1	2 (1)	5 (3)
Carbon	Carbon	1.8 (0.065)	20 (3)	—	<0.1	5–90 (3–50)	2 (1)

Table 4 Properties of Selected Thermosetting and Thermoplastic Matrices

	Density [g/cm³ (Pci)]	Modulus [GPa (Msi)]	Tensile Strength [MPa (ksi)]	Elongation to Break (%)	Thermal Conductivity [W/m · K]	Coefficient of Thermal Expansion [ppm/K (ppm/°F)]
Epoxy[a]	1.1–1.4 (0.040–0.050)	3–6 (0.43–0.88)	35–100 (5–15)	1–6	0.1	60 (33)
Thermosetting polyester[a]	1.2–1.5 (0.043–0.054)	2–4.5 (0.29–0.65)	40–90 (6–13)	2	0.2	100–200 (56–110)
Polypropylene[b]	0.90 (0.032)	1–4 (0.15–0.58)	25–38 (4–6)	>300	0.2	110 (61)
Nylon 6-6[b]	1.14 (0.041)	1.4–2.8 (0.20–0.41)	60–75 (9–11)	40–80	0.2	90 (50)
Polycarbonate[b]	1.06–1.20 (0.038–0.043)	2.2–2.4 (0.32–0.35)	45–70 (7–10)	50–100	0.2	70 (39)
Polysulfone[b]	1.25 (0.045)	2.2 (0.32)	76 (11)	50–100	—	56 (31)
Polyetherimide[b]	1.27 (0.046)	3.3 (0.48)	110 (16)	60	—	62 (34)
Polyamideimide[b]	1.4 (0.050)	4.8 (0.7)	190 (28)	17	—	63 (35)
Polyphenylene sulfide[b]	1.36 (0.049)	3.8 (0.55)	65 (10)	4	—	54 (30)
Polyether etherketone[b]	1.26–1.32 (0.046–0.048)	3.6 (0.52)	93 (13)	50	—	47 (26)

[a] Thermoset.

[b] Thermoplastic.

Epoxies are the workhorse materials for airframe structures and other aerospace applications, with decades of successful flight experience to their credit. They produce composites with excellent structural properties. Epoxies tend to be rather brittle materials, but toughened formulations with greatly improved impact resistance are available. The maximum service temperature is affected by reduced elevated temperature structural properties resulting from water absorption. A typical airframe limit is about 120°C (250°F).

Bismaleimide resins are used for aerospace applications requiring higher temperature capabilities than can be achieved by epoxies. They are employed for temperatures of up to about 200°C (390°F).

Thermosetting polyimides are used for applications with temperatures as high as 250–290°C (500–550°F).

Cyanate ester resins are not as moisture sensitive as epoxies and tend to outgas much less. Formulations with operating temperatures as high as 205°C (400°F) are available.

Thermosetting polyesters are the workhorse resins in commercial applications. They are relatively inexpensive, easy to process, and corrosion resistant.

Vinyl esters are also widely used in commercial applications. They have better corrosion resistance than polyesters but are somewhat more expensive.

Phenolic resins have good high-temperature resistance and produce less smoke and toxic products than most resins when burned. They are used in applications such as aircraft interiors and offshore oil platform structures, for which fire resistance is a key design requirement.

Thermoplastic Resins. Thermoplastics are divided into three main classes: amorphous, crystalline, and liquid crystal. Polycarbonate, acrylonitrile–butadiene–styrene (ABS), polystyrene, polysulfone, and polyetherimide are amorphous materials. Crystalline thermoplastics include nylon, polyethylene, polyphenylene sulfide, polypropylene, acetal, polyethersulfone, and polyether etherketone (PEEK). Amorphous thermoplastics tend to have poor solvent resistance. Crystalline materials tend to be better in this respect. Relatively inexpensive thermoplastics such as nylon are extensively used with chopped E-glass fiber reinforcements in countless injection-molded parts. There are an increasing number of applications using continuous fiber-reinforced thermoplastics.

Metals

The metals initially used for MMC matrix materials generally were conventional alloys. Over time, however, many special matrix materials tailored for use in composites have been developed. The key metallic matrix materials used for structural MMCs are alloys of aluminum, titanium, iron, and intermetallic compounds, such as titanium aluminides. However, many other metals have been used as matrix materials, such as copper, lead, magnesium, cobalt, silver, and superalloys. The *in situ* properties of metals in a composite depend on the manufacturing process and, because metals are elastic–plastic materials, the history of mechanical stresses and temperature changes to which they are subjected.

Ceramic Matrix Materials

The key ceramics used as CMC matrices are silicon carbide, alumina, silicon nitride, mullite, and various cements. The properties of ceramics, especially strength, are even more process sensitive than those of metals. In practice, it is very difficult to determine the *in situ* properties of ceramic matrix materials in a composite.

As discussed earlier, in the section on fiber properties, ceramics are very flaw sensitive, resulting in a decrease in strength with increasing material volume, a phenomenon called ‘‘size effect.’’ As a result, there is no single value that describes the tensile strength of ceramics. In fact, because of the very brittle nature of ceramics, it is difficult to measure tensile strength, and flexural strength (often called modulus of rupture) is typically reported. It should be noted that flexural strength is also dependent on specimen size and is generally much higher than that of a tensile coupon of the same dimensions. In view of the great difficulty in measuring a simple property like tensile strength, which arises from their flaw sensitivity, it is not surprising that monolithic ceramics have had limited success in applications where they are subjected to significant tensile stresses.

The fracture toughness of ceramics is typically in the range of 3–6 $\mathrm{MPa \cdot m^{1/2}}$. Those of transformation-toughened materials are somewhat higher. For comparison, the fracture toughnesses of structural metals are generally greater than 20 $\mathrm{MPa \cdot m^{1/2}}$.

Carbon Matrix Materials

Carbon is a remarkable material. It includes materials ranging from lubricants to diamonds and structural fibers. The forms of carbon matrices resulting from the various carbon/carbon manufacturing processes tend to be rather weak, brittle materials. Some forms have very high thermal conductivities. As for ceramics, *in situ* matrix properties are difficult to measure.

3 PROPERTIES OF COMPOSITE MATERIALS

There are a large and increasing number of materials in all four classes of composites: polymer matrix composites (PMCs), metal matrix composites (MMCs), ceramic matrix composites (CMCs), and carbon/carbon composites (CCCs). In this section, we present mechanical and physical properties of some of the key materials in each class.

Initially, the excellent mechanical properties of composites was the main reason for their use. However, there are an increasing number of applications for which the unique and tailorable physical properties of composites are key considerations. For example, the extremely high thermal conductivity and tailorable coefficient of thermal expansion (CTE) of some composite material systems are leading to their increasing use in electronic packaging. Similarly, the extremely high stiffness, near-zero CTE, and low density of carbon fiber-reinforced polymers have made these composites the materials of choice in spacecraft structures.

Composites are complex, heterogeneous, and often anisotropic material systems. Their properties are affected by many variables, including *in situ* constituent properties; reinforcement form, volume fraction and geometry; properties of the interphase, the region where the reinforcement and matrix are joined (also called the interface); and void content. The process by which the composite is made affects many of these variables. The same matrix material and reinforcements, when combined by different processes, may result in composites with very different properties.

Several other important things must be kept in mind when considering composite properties. For one, most composites are proprietary material systems made by proprietary processes. There are few industry or government specifications for composites, as there are for many monolithic structural metals. However, this is also the case for many monolithic ceramics and polymers, which are widely used engineering materials. Despite their inherently proprietary nature, some widely used composite materials made by a number of manufacturers have similar properties. A notable example is standard-modulus (SM) carbon fiber-reinforced epoxy.

Another critical issue is that properties are sensitive to the test methods by which they are measured, and there are many different test methods used throughout the industry. Further, test results are very sensitive to the skill of the technician performing the test. Because of these factors, it is very common to find significant differences in reported properties of what is nominally the same composite material.

In Section 2, we discussed the issue of size effect, which is the decrease in strength with increasing material volume that is observed in monolithic ceramics key reinforcing fibers. There is some evidence, suggestive but not conclusive, of size effects in composite strength properties, as well. However, if composite strength size effects exist at all, they are much less severe than for fibers by themselves. The reason is that the presence of a matrix results in very different failure mechanisms. However, until the issues are resolved definitively, caution should be used in extrapolating strength data from small coupons to large structures, which may have volumes many orders of magnitude greater.

As mentioned earlier, the properties of composites are very sensitive to reinforcement form, volume fraction, and geometry. This is illustrated in Table 5, which presents the properties of several common types of E-glass fiber-reinforced polyester composites. The reinforcement forms are discontinuous fibers, woven roving (a heavy fabric), and straight, parallel continuous fibers. As we shall see, discontinuous reinforcement is not as efficient as continuous. However, discontinuous fibers allow the composite material to flow during processing, facilitating fabrication of complex molded parts.

The composites using discontinuous fibers are divided into three categories. One is bulk molding compound (BMC), also called dough molding compound, in which fibers are relatively short, about 3–12 mm, and are nominally randomly

Table 5 Effect of Fiber Form and Volume Fraction on Mechanical Properties of E-Glass-Reinforced Polyester[4]

	Bulk Molding Compound	Sheet Molding Compound	Chopped Strand Mat	Woven Roving	Unidirectional Axial	Unidirectional Transverse
Glass content (wt. %)	20	30	30	50	70	70
Tensile modulus GPa (Msi)	9 (1.3)	13 (1.9)	7.7 (1.1)	16 (2.3)	42 (6.1)	12 (1.7)
Tensile strength MPa (ksi)	45 (6.5)	85 (12)	95 (14)	250 (36)	750 (110)	50 (7)

oriented in three dimensions. BMC also has a very high loading of mineral particles, such as calcium carbonate, which are added for a variety of reasons: to reduce dimensional changes from resin shrinkage, to obtain a smooth surface, and to reduce cost, among others. Because it contains both particulate and fibrous reinforcement, BMC can be considered a type of hybrid composite.

The second type of composite is chopped strand mat (CSM), which contains discontinuous fibers, typically about 25 mm long, nominally randomly oriented in two directions. The third material is sheet molding compound (SMC), which contains chopped fibers 25–50 mm in length, also nominally randomly oriented in two dimensions. Like BMC, SMC also contains particulate mineral fillers, such as calcium carbonate and clay.

The first thing to note in comparing the materials in Table 5 is that fiber content, here presented in the form of weight percent, differs considerably for the four materials. This is significant, because, as discussed in Section 2, the strength and stiffness of polyester and most polymer matrices is considerably lower than those of E-glass, carbon, and other reinforcing fibers. Composites reinforced with randomly oriented fibers tend to have lower volume fractions than those made with aligned fibers or fabrics. There is a notable exception to this. Some composites with discontinuous-fiber reinforcement are made by chopping up composites reinforced with aligned continuous fibers or fabrics that have high-fiber contents.

Examination of Table 5 shows that the modulus of SMC is considerably greater than that of CSM, even though both have the same fiber content. This is because SMC also has particulate reinforcement. Note, however, that although the particles improve modulus, they do not increase strength. This is generally the case for particle-reinforced polymers, but, as we will see later, particles often do enhance the strengths of MMCs and CMCs, as well as their moduli.

We observe that the modulus of the BMC composite is greater than that of CSM and SMC, even though the former has a much lower fiber content. Most likely, this results from the high-mineral content and also the possibility that the fibers are oriented in the direction of test, and are not truly random. Many processes, especially those involving material flow, tend to orient fibers in one or more preferred directions. If so, then one would find the modulus of the BMC to be much lower than the one presented in the table if measured in other directions. This illustrates one of the limitations of using discontinuous fiber reinforcement: it is often difficult to control fiber orientation.

The moduli and strengths of the composites reinforced with fabrics and aligned fibers are much higher than those with discontinuous fibers, when the former two types of materials are tested parallel to fiber directions. For example, the tensile strength of woven roving is more than twice that of CSM. The properties presented are measured parallel to the warp direction of the fabric (the warp direction is the lengthwise direction of the fabric). The elastic and strength properties in the fill direction, perpendicular to the warp, typically are similar to, but somewhat lower than, those in the warp direction. Here, we assume that the fabric is ‘‘balanced,’’ which means that the number of fibers in the warp and fill directions per unit length are approximately equal. Note, however, that the elastic modulus, tensile strength, and compressive strength at 45° to the warp and fill directions of a fabric are much lower than the corresponding values in the warp and fill directions. This is discussed further in the sections that cover design.

As Table 5 shows, the axial modulus and tensile strength of the unidirectional composite are much greater than those of the fabric. However, the modulus and strength of the unidirectional composite in the transverse direction are considerably lower than the corresponding axial properties. Further, the transverse strength is considerably lower than that of SMC and CSM. In general, the strength of PMCs is weak in directions for which there are no fibers. The low transverse moduli and strengths of unidirectional PMCs are commonly overcome by use of laminates with fibers in several directions. Low through-thickness strength can be improved by use of three-dimensional reinforcement forms. Often, the designer simply assures that through-thickness stresses are within the capability of the material.

In this section, we present representative mechanical and physical properties of key composite materials of interest for a broad range of mechanical engineering applications. The properties represent a distillation of values from many sources. Because of space limitations, it is necessary to be selective in our choice of materials and properties presented. It is simply not possible to present a complete set of data that will cover every possible application. As discussed earlier, there are many textile forms, such as woven fabrics, used as reinforcements. However, we concentrate on aligned, continuous fibers because they produce the highest strength and stiffness. To do a thorough evaluation of composites, the design engineer should consider alternative reinforcement forms. Unless otherwise stated, room temperature property values are presented. We consider mechanical properties in Section 3.1 and physical properties in Section 3.2.

3.1 Mechanical Properties of Composite Materials

In this section, we consider mechanical properties of key PMCs, MMCs, CMCs, and CCCs that are of greatest interest for mechanical engineering applications.

Mechanical Properties of Polymer Matrix Composites

As discussed earlier, polymers are relatively weak, low-stiffness materials. To obtain materials with mechanical properties that are acceptable for structural applications, it is necessary to reinforce them with continuous or discontinuous fibers. The addition of ceramic or metallic particles to polymers results in materials that have increased modulus, but, as a rule, strength typically does not increase significantly, and may actually decrease. However, there are many particle-reinforced polymers used in electronic packaging, primarily because of their physical properties. For these applications, ceramic particles, such as alumina, aluminum nitride, boron nitride, and even diamond, are added to obtain an electrically insulating material with higher thermal conductivity and lower CTE than the monolithic base polymer. Metallic particles such as silver and aluminum are added to create materials that are both electrically and thermally conductive. These materials have replaced lead-based solders in many applications. There are also magnetic composites made by incorporating ferrous or permanent magnet particles in various polymers. A common example is magnetic tape used to record audio and video.

We focus on composites reinforced with continuous fibers because they are the most efficient structural materials. Table 6 presents room temperature mechanical properties of unidirectional polymer matrix composites reinforced with key fibers: E-glass, aramid, boron, standard-modulus (SM) PAN (polyacrilonitrile) carbon, ultrahigh-strength (UHS) PAN carbon, ultrahigh-modulus (UHM) PAN carbon, ultrahigh-modulus (UHM) pitch carbon, and ultrahigh-thermal conductivity (UHK) pitch carbon. We assume that the fiber volume fraction is 60%, a typical value. As discussed in Section 2, UHS PAN carbon is the strongest type of intermediate-modulus (IM) carbon fiber.

The properties presented in Table 6 are representative of what can be obtained at room temperature with a well-made PMC employing an epoxy matrix. Epoxies are widely used, provide good mechanical properties, and can be considered a reference matrix material. Properties of composites using other resins may differ from these, and have to be examined on a case-by-case basis.

The properties of PMCs, especially strengths, depend strongly on temperature. The temperature dependence of polymer properties differs considerably. This is also true for different epoxy formulations, which have different cure and glass transition temperatures. Some polymers, such as polyimides, have good elevated temperature properties that allow them to compete with titanium. There are aircraft gas turbine engine components employing polyimide matrices that see service temperatures as high as 290°C (550°F). Here again, the effect of temperature on composite properties has to be considered on a case-by-case basis.

The properties shown in Table 6 are axial, transverse and shear moduli, Poisson's ratio, tensile and compressive strengths in the axial and transverse directions, and inplane shear strength. The Poisson's ratio presented is called the major Poisson's ratio. It is defined as the ratio of the magnitude of transverse strain divided by axial strain when the composite is loaded in the axial direction. Note that transverse moduli and strengths are much lower than corresponding axial values.

As discussed in Section 2, carbon fibers display nonlinear stress–strain behavior. Their moduli increase under increasing tensile stress and decrease under increasing compressive stress. This makes the method of calculating modulus critical. Various tangent and secant definitions are used throughout the industry, contributing to the confusion in reported properties. The values presented in Table 6, which are approximate, are based on tangents to the stress–strain curves at the origin. Using this definition, tensile and compressive moduli are usually very similar. However, this is not the case for moduli using various secant definitions. Using these definitions typically produces compression moduli that are significantly lower than tension moduli.

Because of the low transverse strengths of unidirectional laminates, they are rarely used in structural applications. The design engineer uses laminates with layers in several directions to meet requirements for strength, stiffness, buckling, and so on. There are an infinite number of laminate geometries that can be selected. For comparative purposes, it is useful to consider quasi-isotropic laminates, which have the same elastic properties in all directions in the plane. Laminates are quasi-isotropic when they have the same percentage of layers every $180/n°$, where $n \geq 3$. The most common quasi-isotropic laminates have layers that repeat every 60°, 45°, or 30°. We note, however, that strength prop-

Table 6 Mechanical Properties of Selected Unidirectional Polymer Matrix Composites

Fiber	Axial Modulus [GPa (Msi)]	Transverse Modulus [GPa (Msi)]	Inplane Shear Modulus [GPa (Msi)]	Poisson's Ratio	Axial Tensile Strength [MPa (ksi)]	Transverse Tensile Strength [MPa (ksi)]	Axial Compressive Strength [MPa (ksi)]	Transverse Compressive Strength [MPa (ksi)]	Inplane Shear Strength [MPa (ksi)]
E-glass	45 (6.5)	12 (1.8)	5.5 (0.8)	0.28	1020 (150)	40 (7)	620 (90)	140 (20)	70 (10)
Aramid	76 (11)	5.5 (0.8)	2.1 (0.3)	0.34	1240 (180)	30 (4.3)	280 (40)	140 (20)	60 (9)
Boron	210 (30)	19 (2.7)	4.8 (0.7)	0.25	1240 (180)	70 (10)	3310 (480)	280 (40)	90 (13)
SM carbon (PAN)	145 (21)	10 (1.5)	4.1 (0.6)	0.25	1520 (220)	41 (6)	1380 (200)	170 (25)	80 (12)
UHS carbon (PAN)	170 (25)	10 (1.5)	4.1 (0.6)	0.25	3530 (510)	41 (6)	1380 (200)	170 (25)	80 (12)
UHM carbon (PAN)	310 (45)	9 (1.3)	4.1 (0.6)	0.20	1380 (200)	41 (6)	760 (110)	170 (25)	80 (12)
UHM carbon (pitch)	480 (70)	9 (1.3)	4.1 (0.6)	0.25	900 (130)	20 (3)	280 (40)	100 (15)	41 (6)
UHK carbon (pitch)	480 (70)	9 (1.3)	4.1 (0.6)	0.25	900 (130)	20 (3)	280 (40)	100 (15)	41 (6)

erties in the plane are not isotropic for these laminates, although they tend to become more uniform as the angle of repetition becomes smaller.

Table 7 presents the mechanical properties of quasi-isotropic laminates. Note that the moduli and strengths are much lower than the axial properties of unidirectional laminates made of the same material. In most applications, laminate geometry is such that the maximum axial modulus and tensile and compressive strengths fall somewhere between axial unidirectional and quasi-isotropic values.

The tension–tension fatigue behavior of unidirectional composites, discussed in Section 1, is one of their great advantages over metals (Fig. 6). In general the tension–tension *S–N* curves (curves of maximum stress plotted as a function of cycles to failure) of PMCs reinforced with carbon, boron, and aramid fibers are relatively flat. Glass-fiber-reinforced composites show a greater reduction in strength with increasing number of cycles. Still, PMCs reinforced with HS glass are widely used in applications for which fatigue resistance is a critical design consideration, such as helicopter rotors.

Metals are more likely to fail in fatigue when subjected to fluctuating tensile rather than compressive load. This is because they tend to fail by crack propagation under fatigue loading. However, the failure modes in composites are very different and more complex. One consequence is that composites tend to be more susceptible to fatigue failure when loaded in compression. Figure 6 shows the cycles to failure as a function of maximum stress for carbon-fiber-reinforced epoxy laminates subjected to tension–tension and compression–compression fatigue. The laminates have 60% of their layers oriented at 0°, 20% at +45°, and 20% at −45°. They are subjected to a fluctuating load in the 0° direction. The ratios of minimum stress-to-maximum stress (R) for tensile and compressive fatigue are 0.1 and 10, respectively. We observe that the reduction in strength is much greater for compression–compression fatigue. However, the composite compressive fatigue strength at 10^7 cycles is still considerably greater than the corresponding tensile value for aluminum.

Polymer matrix composites reinforced with carbon and boron are very resistant to deformation and failure under sustained static load when they are loaded in a fiber-dominated direction. (These phenomena are called creep and creep rupture, respectively.) The creep and creep rupture behavior of aramid is not quite as good. Glass fibers display significant creep, and creep rupture is an important design consideration. Polymers are viscoelastic materials that typically display significant creep when they are not constrained with fibers. Therefore, creep should be considered when composites are subjected to significant stresses in matrix-dominated directions, such as the laminate through-thickness direction.

Mechanical Properties of Metal Matrix Composites

Monolithic metallic alloys are the most widely used materials in mechanical engineering applications. By reinforcing them with continuous fibers, discontinuous fibers, whiskers and particles, we create new materials with enhanced or modified properties, such as higher strength and stiffness, better wear resistance, lower CTE, and so on. In some cases, the improvements are dramatic.

The greatest increases in strength and modulus are achieved with continuous fibers. However, the relatively high cost of many continuous reinforcing fibers used in MMCs has limited the application of these materials. The most widely

Table 7 Mechanical Properties of Selected Quasi-Isotropic Polymer Matrix Composites

Fiber	Axial Modulus [GPa (Msi)]	Transverse Modulus [GPa (Msi)]	Inplane Shear Modulus [GPa (Msi)]	Poisson's Ratio	Axial Tensile Strength [MPa (ksi)]	Transverse Tensile Strength [MPa (ksi)]	Axial Compressive Strength [MPa (ksi)]	Transverse Compressive Strength [MPa (ksi)]	Inplane Shear Strength [MPa (ksi)]
E-glass	23 (3.4)	23 (3.4)	9.0 (1.3)	0.28	550 (80)	550 (80)	330 (48)	330 (48)	250 (37)
Aramid	29 (4.2)	29 (4.2)	11 (1.6)	0.32	460 (67)	460 (67)	190 (28)	190 (28)	65 (9.4)
Boron	80 (11.6)	80 (11.6)	30 (4.3)	0.33	480 (69)	480 (69)	1100 (160)	1100 (160)	360 (52)
SM carbon (PAN)	54 (7.8)	54 (7.8)	21 (3.0)	0.31	580 (84)	580 (84)	580 (84)	580 (84)	410 (59)
UHS carbon (PAN)	63 (9.1)	63 (9.1)	21 (3.0)	0.31	1350 (200)	1350 (200)	580 (84)	580 (84)	410 (59)
UHM carbon (PAN)	110 (16)	110 (16)	41 (6.0)	0.32	490 (71)	490 (71)	270 (39)	70 (39)	205 (30)
UHM carbon (pitch)	165 (24)	165 (24)	63 (9.2)	0.32	310 (45)	310 (45)	96 (14)	96 (14)	73 (11)
UHK carbon (pitch)	165 (24)	165 (24)	63 (9.2)	0.32	310 (45)	310 (45)	96 (14)	96 (14)	73 (11)

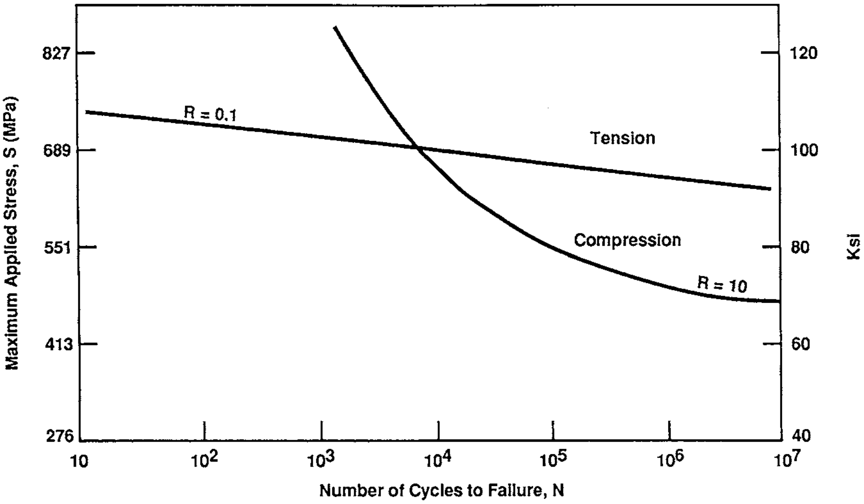


Fig. 6 Cycles to failure as a function of maximum stress for carbon-fiber-reinforced epoxy laminates loaded in tension–tension ($R = 0.1$) and compression–compression ($R = -10$) fatigue (after Ref. 5).

used MMCs are reinforced with discontinuous fibers or particles. This may change as new, lower cost continuous fibers and processes are developed and as cost drops with increasing production volume.

Continuous Fiber-Reinforced MMCs. One of the major advantages of MMCs reinforced with continuous fibers over PMCs is that many, if not most, unidirectional MMCs have much greater transverse strengths, which allow them to be used in a unidirectional configuration. Table 8 presents representative mechanical properties of selected unidirectional MMCs reinforced with continuous fibers corresponding to a nominal fiber volume fraction of 50%. The values represent a distillation obtained from numerous sources. In general, the axial moduli of the composites are much greater than those of the monolithic base metals used for the matrices. However, MMC transverse strengths are typically lower than those of the parent matrix materials.

Mechanical Properties of Discontinuous Fiber-Reinforced MMCs. One of the primary mechanical engineering applications of discontinuous fiber-reinforced MMCs is in internal combustion engine components. Fibers are added primarily to improve the wear resistance and elevated temperature strength and fatigue properties of aluminum. The improvement in wear resistance eliminates the need for cast-iron sleeves in engine blocks and cast-iron insert rings in pistons. Fiber-reinforced aluminum composites also have higher thermal conductivities than cast iron and, when fiber volume fractions are relatively low, their CTEs are closer to that of unreinforced aluminum, reducing thermal stresses.

The key reinforcements used in internal combustion engine components to increase wear resistance are discontinuous alumina and alumina–silica fibers. In one application, Honda Prelude engine blocks, carbon fibers are combined with

Table 8 Mechanical Properties of Selected Unidirectional Continuous Fiber-Reinforced Metal Matrix Composites

Fiber	Matrix	Density [g/cm^3 (Pci)]	Axial Modulus [GPa (Msi)]	Transverse Modulus [GPa (Msi)]	Axial Tensile Strength [MPa (ksi)]	Transverse Tensile Strength [MPa (ksi)]	Axial Compressive Strength [MPa (ksi)]
UHM carbon (pitch)	Aluminum	2.4 (0.090)	450 (65)	15 (5)	690 (100)	15 (5)	340 (50)
Boron	Aluminum	2.6 (0.095)	210 (30)	140 (20)	1240 (180)	140 (20)	1720 (250)
Alumina	Aluminum	3.2 (0.12)	240 (35)	130 (19)	1700 (250)	120 (17)	1800 (260)
Silicon carbide	Titanium	3.6 (0.13)	260 (38)	170 (25)	1700 (250)	340 (50)	2760 (400)

alumina to tailor both wear resistance and coefficient of friction of cylinder walls. Wear resistance is not an inherent property, so that there is no single value that characterizes a material. However, in engine tests, it was found that ring groove wear for an alumina fiber-reinforced aluminum piston was significantly less than that for one with a cast-iron insert.

Mechanical Properties of Particle-Reinforced MMCs. Particle-reinforced metals are a particularly important class of MMCs for engineering applications. A wide range of materials fall into this category, and a number of them have been used for many years. An important example is a material consisting of tungsten carbide particles embedded in a cobalt matrix that is used extensively in cutting tools and dies. This composite, often referred to as a cermet, cemented carbide, or simply, but incorrectly, ''tungsten carbide,'' has much better fracture toughness than monolithic tungsten carbide, which is a brittle ceramic material. Another interesting MMC, tungsten carbide particle-reinforced silver, is a key circuit breaker contact pad material. Here, the composite provides good electrical conductivity and much greater hardness and wear resistance than monolithic silver, which is too soft to be used in this application. Ferrous alloys reinforced with titanium carbide particles, discussed in the next subsection, have been used for many years in commercial applications. Compared to the monolithic base metals, they offer greater wear resistance and stiffness and lower density.

Mechanical Properties of Titanium Carbide Particle-Reinforced Steel. A number of ferrous alloys reinforced with titanium carbide particles have been used in mechanical system applications for many years. To illustrate the effect of the particulate reinforcements, we consider a particular composite consisting of austenitic stainless steel reinforced with 45% by volume of titanium carbide particles. The modulus of the composite is 304 GPa (44 Msi) compared to 193 GPa (28 Msi) for the monolithic base metal. The specific gravity of the composite is 6.45, about 20% lower than that of monolithic matrix, 8.03. The specific stiffness of the composite is almost double that of the unreinforced metal.

Mechanical Properties of Silicon Carbide Particle-Reinforced Aluminum. Aluminum reinforced with silicon carbide particles is one of the most important of the newer types of MMCs. A wide range of materials fall into this category. They are made by a variety of processes. Properties depend on the type of particle, particle volume fraction, matrix alloy, and the process used to make them. Table 9 shows how representative composite properties vary with particle volume fraction. In general, as particle volume fraction increases, modulus and yield strength increase and fracture toughness and tensile ultimate strain decrease. Particle reinforcement also improves short-term elevated temperature strength properties and fatigue resistance.

Mechanical Properties of Alumina Particle-Reinforced Aluminum. Alumina particles are used to reinforce aluminum as an alternative to silicon carbide particles because they do not react as readily with the matrix at high temperatures and are less expensive. Consequently, alumina-reinforced composites can be used in a wider range of processes and applications. However, the stiffness and thermal conductivity of alumina are lower than the corresponding properties of silicon carbide, and these characteristics are reflected in somewhat lower values for composite properties.

Table 9 Mechanical Properties of Silicon Carbide Particle-Reinforced Aluminum

Property	Aluminum (6061–T6)	Titanium (6Al–4V)	Steel (4340)	Composite Particle Volume Fraction		
				25	55	70
Modulus, GPa (Msi)	69 (10)	113 (16.5)	200 (29)	114 (17)	186 (27)	265 (38)
Tensile yield strength, MPa (ksi)	275 (40)	1000 (145)	1480 (215)	400 (58)	495 (72)	225 (33)
Tensile ultimate strength, MPa (ksi)	310 (45)	1100 (160)	1790 (260)	485 (70)	530 (77)	225 (33)
Elongation (%)	15	5	10	3.8	0.6	0.1
Density, g/cm^3 (lb/in.^{3})	2.77 (0.10)	4.43 (0.16)	7.76 (0.28)	2.88 (0.104)	2.96 (0.107)	3.00 (0.108)
Specific modulus, GPa	5	26	26	40	63	88

Mechanical Properties of Ceramic Matrix Composites

Ceramics, in general, are characterized by high stiffness and hardness, resistance to wear, corrosion and oxidation, and high-temperature operational capability. However, they also have serious deficiencies that have severely limited their use in applications that are subjected to significant tensile stresses. Ceramics have very low fracture toughness, which makes them very sensitive to the presence of small flaws. This results in great strength scatter and poor resistance to thermal and mechanical shock. Civil engineers recognized this deficiency long ago and, in construction, ceramic materials like stone and concrete are rarely used to carry tensile loads. In concrete, this function has been relegated to reinforcing bars made of steel or, more recently, PMCs. An important exception has been in lightly loaded structures where dispersed reinforcing fibers of asbestos, steel, glass, and carbon allow modest tensile stresses to be supported.

In CMCs, fibers, whiskers, and particles are combined with ceramic matrices to improve fracture toughness, which reduces strength scatter and improves thermal and mechanical shock resistance. By a wide margin, the greatest increases in fracture resistance result from the use of continuous fibers. Table 10 compares fracture toughnesses of structural metallic alloys with those of monolithic ceramics and CMCs reinforced with whiskers and continuous fibers. The low fracture toughness of monolithic ceramics gives rise to very small critical flaw sizes. For example, the critical flaw sizes for monolithic ceramics corresponding to a failure stress of 700 MPa (about 100 ksi) are in the range of 20–80 μm. Flaws of this size are difficult to detect with conventional nondestructive techniques.

The addition of continuous fibers to ceramics can, if done properly, significantly increase the effective fracture toughness of ceramics. For example, as Table 10 shows, addition of silicon carbide fibers to a silicon carbide matrix results in a CMC having a fracture toughness in the range of aluminum alloys.

The addition of continuous fibers to a ceramic matrix also changes the failure mode. Figure 7 compares the tensile stress–strain curves for a typical monolithic ceramic and a conceptual continuous fiber-reinforced CMC. The monolithic material has a linear stress–strain curve and fails catastrophically at a low strain level. However, the CMC displays a nonlinear stress–strain curve with much more area under the curve, indicating that more energy is absorbed during failure

Table 10 Fracture Toughness of Structural Alloys, Monolithic Ceramics, and Ceramic Matrix Composites

Matrix	Reinforcement	Fracture Toughness MPa $m^{1/2}$
Aluminum	none	30–45
Steel	none	40–65[a]
Alumina	none	3–5
Silicon carbide	none	3–4
Alumina	Zirconia particles[b]	6–15
Alumina	Silicon carbide whiskers	5–10
Silicon carbide	Continuous silicon carbide fibers	25–30

[a] The toughness of some alloys can be much higher.

[b] Transformation-toughened.

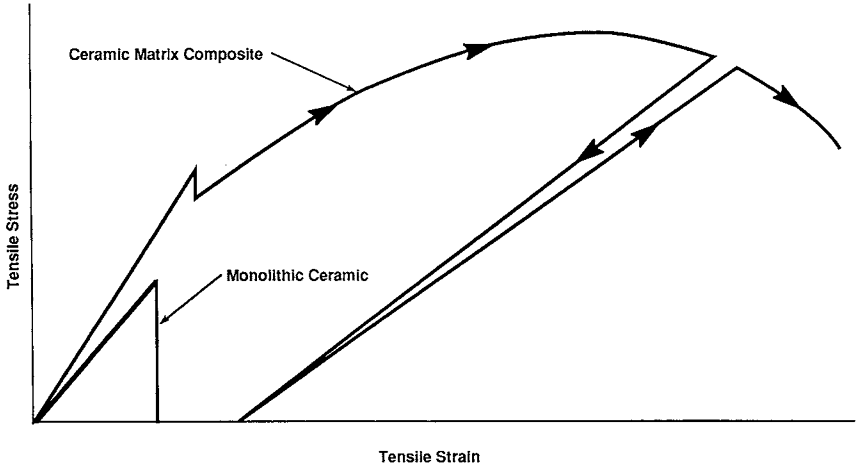


Fig. 7 Stress–strain curves for a monolithic ceramic and ceramic matrix composite reinforced with continuous fibers.

and that the material has a less catastrophic failure mode. The fiber–matrix interphase properties must be carefully tailored and maintained over the life of the composite to obtain this desirable behavior.

Although the CMC stress–strain curve looks, at first, like that of an elastic–plastic metal, this is deceiving. The departure from linearity in the CMC results from internal damage mechanisms, such as the formation of microcracks in the matrix. The fibers bridge the cracks, preventing them from propagating. However, the internal damage is irreversible. As the figure shows, the slope of the stress–strain curve during unloading and subsequent reloading is much lower than that representing initial loading. For an elastic–plastic material, the slopes of the unloading and reloading curves are parallel to the initial elastic slope.

There are numerous CMCs at various stages of development. One of the most mature types consists of a silicon carbide matrix reinforced with fabric woven of silicon-carbide-based fibers. These composites are commonly referred to as SiC/SiC. We consider one version. Because the modulus of the particular silicon-carbide-based fibers used in this material is lower than that of pure silicon carbide, the modulus of the composite, about 210 GPa (30 Msi), is lower than that of monolithic silicon carbide, 440 GPa (64 Msi). The flexural strength of the composite parallel to the fabric warp direction, about 300 MPa (44 ksi), is maintained to a temperature of at least 1100°C for short times. Long-term strength behavior depends on degradation of the fibers, matrix, and interphase. Because of the continuous fiber reinforcement, SiC/SiC displays excellent resistance to severe thermal shock.

Mechanical Properties of Carbon/Carbon Composites

Carbon/carbon composites consist of continuous and discontinuous carbon fibers embedded in carbon matrices. As for other composites, there are a wide range of materials that fall in this category. The variables affecting properties include type of fiber, reinforcement form, and volume fraction and matrix characteristics.

Historically, CCCs were first used because of their excellent resistance to high-temperature ablation. Initially, strengths and stiffnesses were low, but these properties have steadily increased over the years. CCCs are an important class of materials in high-temperature applications such as aircraft brakes, rocket nozzles, racing car brakes and clutches, glass-making equipment, and electronic packaging, among others.

One of the most significant limitations of CCCs is oxidation, which begins at a temperature threshold of approximately 370°C (700°F) for unprotected materials. Addition of oxidation inhibitors raises the threshold substantially. In inert atmospheres, CCCs retain their properties to temperatures as high as 2800°C (5000°F).

Carbon matrices are typically weak, brittle, low-stiffness materials. As a result, transverse and through-thickness elastic moduli and strength properties of unidirectional CCCs are low. Because of this, two-dimensional and three-dimensional reinforcement forms are commonly used. In the direction of fibrous reinforcement, it is possible to obtain moduli as high as 340 GPa (50 Msi), tensile strengths as high as 700 MPa (100 ksi), and compressive strengths as high as 800 MPa (110 ksi). In directions orthogonal to fiber directions, elastic moduli are in the range of 10 MPa (1.5 ksi), tensile strengths 14 MPa (2 ksi), and compressive strengths 34 MPa (5 ksi).

3.2 Physical Properties of Composite Materials

Material physical properties are critical for many applications. In this category, we include, among others, density, CTE, thermal conductivity, and electromagnetic characteristics. In this section, we concentrate on the properties of most general interest to mechanical engineers: density, CTE, and thermal conductivity.

Thermal control is a particularly important consideration in electronic packaging because failure rates of semiconductors increase exponentially with temperature. Since conduction is an important method of heat removal, thermal conductivity is a key material property. For many applications, such as spacecraft, aircraft, and portable systems, weight is also an important factor, and consequently, material density is also significant. A useful figure of merit is specific thermal conductivity, defined as thermal conductivity divided by density. Specific thermal conductivity is analogous to specific modulus and specific strength.

In addition to thermal conductivity and density, CTE is also of great significance in many applications. For example, semiconductors and ceramic substrates used in electronics are brittle materials with coefficients of expansion in the range of about 3–7 ppm/K. Semiconductors and ceramic substrates are typically attached to supporting components, such as packages, printed circuit boards (PCBs), and heat sinks with solder or an adhesive. If the CTE of the supporting material is significantly different from that of the ceramic or semiconductor, thermal stresses arise when the assembly is subjected to a change in temperature. These stresses can result in failure of the components or the joint between them.

A great advantage of composites is that there are an increasing number of material systems that combine high thermal conductivity with tailorable CTE, low density, and excellent mechanical properties. Composites can truly be called multifunctional materials.

The key composite materials of interest for thermal control are PMCs, MMCs, and CCCs reinforced with ultrahigh-thermal conductivity (UHK) carbon fibers, which are made from pitch; silicon carbide particle-reinforced aluminum; beryllium oxide particle-reinforced beryllium; and diamond particle-reinforced aluminum and copper. There also are a number of other special CCCs developed specifically for thermal control applications.

Table 11 presents physical properties of a variety of unidirectional composites reinforced with UHK carbon fibers, along with those of monolithic copper and 6063 aluminum for comparison. Unidirectional composites are useful for directing heat in a particular direction. The particular fibers represented have a nominal axial thermal conductivity of 1100 W/m · K. Predicted properties are shown for four matrices: epoxy, aluminum, copper, and carbon. Typical reinforcement volume fractions (V/O) are assumed. As Table 11 shows, the specific axial thermal conductivities of the composites are significantly greater than those of aluminum and copper.

Figure 8 presents thermal conductivity as a function of CTE for various materials used in electronic packaging. Materials shown include silicon (Si) and gallium arsenide (GaAs) semiconductors; alumina (Al_2O_3), beryllium oxide (BeO), and aluminum nitride (AlN) ceramic substrates; and monolithic aluminum, beryllium, copper, silver, and Kovar, a nickel–iron alloy. Other monolithic materials included are diamond and pyrolitic graphite, which have very high thermal conductivities in some forms. The figure also presents metal–metal composites, such as copper–tungsten (Cu–W), copper–molybdenum (Cu–Mo), beryllium–aluminum (Be–Al), aluminum–silicon (Al–Si), and Silvar, which contains silver and a nickel–iron alloy. The latter materials can be considered composites rather than true alloys because the two components have low solubility and appear as distinct phases at room temperature.

As Figure 8 shows, aluminum, copper, and silver have relatively high thermal conductivities but have CTEs much greater than desirable for most electronic packaging applications. By combining these metals with various reinforcements, it is possible to create new materials having CTEs isotropic in two dimensions (quasi-isotropic) or three dimensions in the desired range. The figure shows a number of composites: copper reinforced with UHK carbon fibers (C/Cu), aluminum reinforced with UHK carbon fibers (C/Al), carbon reinforced with UHK carbon fibers (C/C), epoxy reinforced with UHK carbon fibers (C/Ep), aluminum reinforced with silicon carbide particles [(SiC)p/Al], beryllium oxide particle-reinforced beryllium [(BeO)p/Be], diamond particle-reinforced copper [(Diamond)p/Cu], and E-glass fiber-reinforced epoxy (E-glass/Ep). With the exception of E-glass/Ep, C/Ep, and C/C, all of the composites have some configurations with CTEs in the desired range. The thermal conductivities of the composites presented are generally similar to, or better than, that of aluminum, while their CTEs are much closer to the goal range of 3–7 ppm/K. E-glass/Ep is an exception.

Note that although the CTEs of C/Ep and C/C are lower than desired for electronic packaging applications, the differences between their CTEs and those of ceramics and semiconductors are much less than the differences for aluminum and copper. Consequently, use of the composites can result in lower thermal stresses for a given temperature change.

Table 11 Physical Properties of Selected Unidirectional Composites and Monolithic Metals

Matrix	Reinforcement	V/O (%)	Density [g/cm^3 (Pci)]	Axial Coefficient of Thermal Expansion [ppm/K (ppm/°F)]	Axial Thermal Conductivity [W/m · K (Btu/h · ft · °F)]	Transverse Thermal Conductivity [W/m · K (Btu/h · ft · °F)]	Specific Axial Thermal Conductivity [W/m · K (Btu/h · ft · °F)]
Aluminum (6063)	—	—	2.7 (0.098)	23 (13)	218 (126)	218 (126)	81
Copper	—	—	8.9 (0.32)	17 (9.8)	400 (230)	400 (230)	45
Epoxy	UHK carbon fibers	60	1.8 (0.065)	−1.2 (−0.7)	660 (380)	2 (1.1)	370
Aluminum	UHK carbon fibers	50	2.45 (0.088)	−0.5 (−0.3)	660 (380)	50 (29)	110
Copper	UHK carbon fibers	50	5.55 (0.20)	−0.5 (−0.3)	745 (430)	140 (81)	130
Carbon	UHK carbon fibers	40	1.85 (0.067)	−1.5 (−0.8)	740 (430)	45 (26)	400

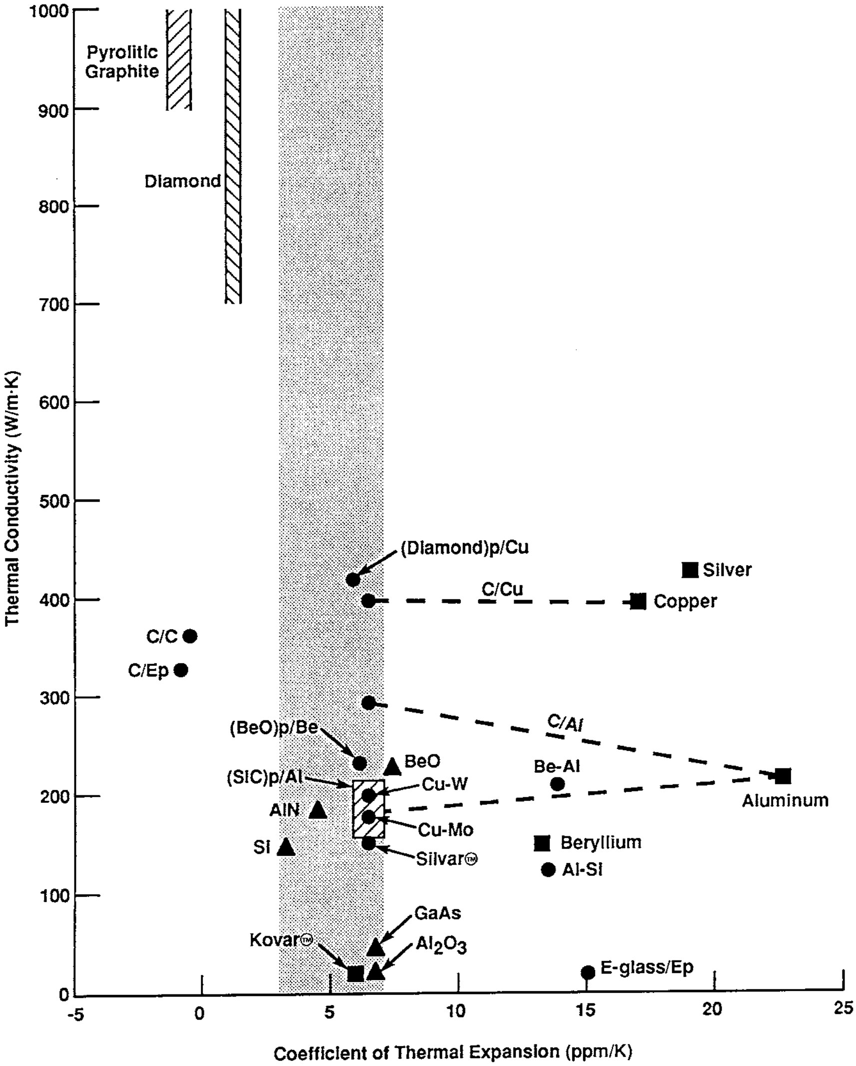


Fig. 8 Thermal conductivity as a function of coefficient of thermal expansion for selected monolithic materials and composites used in electronic packaging.

The physical properties of the materials shown in Figure 8 and others are presented in Table 12.

The advantages of composites are even greater than those of conventional packaging materials when weight is considered. Figure 9 presents the specific thermal conductivities and CTEs of the materials appearing in Figure 8. Here, we find order-of-magnitude improvements. As discussed earlier, when a critical property is increased by an order of magnitude it tends to have a revolutionary effect on technology. Several composites demonstrate this level of improvement; as a result, composites are being used in an increasing number of electronic packaging and thermal control applications.

Table 12 Physical Properties of Isotropic and Quasi-Isotropic Composites and Monolithic Materials Used in Electronic Packaging

Matrix	Reinforcement	V/O (%)	Density [g/cm^3 (Pci)]	Coefficient of Thermal Expansion [ppm/K (ppm/°F)]	Thermal Conductivity [W/m · K (Btu/h · ft · °F)]	Specific Thermal Conductivity [W/m · K]
Aluminum (6063)	—	—	2.7 (0.098)	23 (13)	218 (126)	81
Copper	—	—	8.9 (0.32)	17 (9.8)	400 (230)	45
Beryllium	—	—	1.86 (0.067)	13 (7.2)	150 (87)	81
Magnesium	—	—	1.80 (0.065)	25 (14)	54 (31)	12
Titanium	—	—	4.4 (0.16)	9.5 (5.3)	16 (9.5)	4
Stainless steel (304)	—	—	8.0 (0.29)	17 (9.6)	16 (9.4)	2
Molybdenum	—	—	10.2 (0.37)	5.0 (2.8)	140 (80)	14
Tungsten	—	—	19.3 (0.695)	4.5 (2.5)	180 (104)	9
Invar	—	—	8.0 (0.29)	1.6 (0.9)	10 (6)	1
Kovar	—	—	8.3 (0.30)	5.9 (3.2)	17 (10)	2
Alumina (99% pure)	—	—	3.9 (0.141)	6.7 (3.7)	20 (12)	5
Beryllia	—	—	2.9 (0.105)	6.7 (3.7)	250 (145)	86
Aluminum nitride	—	—	3.2 (0.116)	4.5 (2.5)	250 (145)	78
Silicon	—	—	2.3 (0.084)	4.1 (2.3)	150 (87)	65
Gallium arsenide	—	—	5.3 (0.19)	5.8 (3.2)	44 (25)	8
Diamond	—	—	3.5 (0.13)	1.0 (0.6)	2000 (1160)	570
Pyrolitic graphite	—	—	2.3 (0.083)	−1 (−0.6)	1700 (980)	750
Aluminum–silicon	—	—	2.5 (0.091)	13.5 (7.5)	126 (73)	50
Beryllium–aluminum	—	—	2.1 (0.076)	13.9 (7.7)	210 (121)	100
Copper–tungsten (10/90)	—	—	17 (0.61)	6.5 (3.6)	209 (121)	12
Copper–molybdenum (15/85)	—	—	10 (0.36)	6.6 (3.7)	184 (106)	18
Aluminum	SiC particles	70	3.0 (0.108)	6.5 (3.6)	190 (110)	63
Beryllium	BeO particles	60	2.6 (0.094)	6.1 (3.4)	240 (139)	92
Copper	Diamond particles	55	5.9 (0.21)	5.8 (3.2)	420 (243)	71
Epoxy	UHK carbon fibers	60	1.8 (0.065)	−0.7 (−0.4)	330 (191)	183
Aluminum	UHK carbon fibers	26	2.6 (0.094)	6.5 (3.6)	290 (168)	112
Copper	UHK carbon fibers	26	7.2 (0.26)	6.5 (3.6)	400 (230)	56
Carbon	UHK carbon fibers	40	1.8 (0.065)	−1 (−0.6)	360 (208)	195

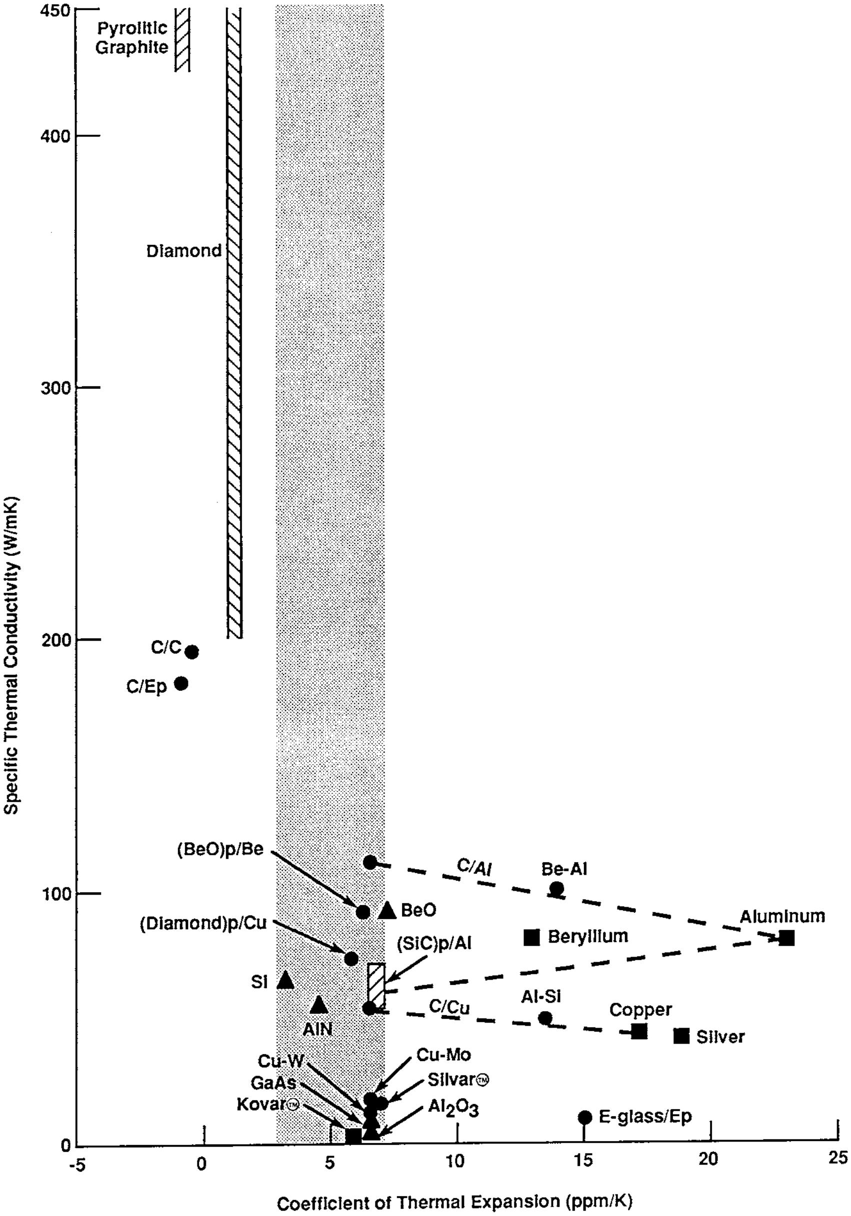


Fig. 9 Specific thermal conductivity (thermal conductivity divided by specific gravity) for selected monolithic materials and composites used in electronic packaging.

Physical Properties of Polymer Matrix Composites

Table 13 presents physical properties of the polymer matrix composites discussed in Section 3.1. A fiber volume fraction of 60% is assumed. The densities of all of the materials are considerably lower than that of aluminum, and some are lower than that of magnesium. This reflects the low densities of both fibers and matrix materials. The low densities of most polymers give PMCs a significant advantage over most MMCs and CMCs, all other things being equal.

We observe that all of the composites have relatively low-axial CTEs. This results from the combination of low-fiber-axial CTE, high fiber stiffness, and low matrix stiffness. Note that the axial CTEs of PMCs reinforced with aramid fibers and some carbon fibers are negative. This means that, contrary to the general behavior of most monolithic materials, they contract when heated. The transverse CTEs of the composites are all positive, and much larger than the magnitudes of the corresponding axial CTEs. This results from the high CTE of the matrix and a Poisson effect caused by constraint of the matrix in the axial direction and lack of constraint in the transverse direction. The transverse CTE of aramid composites is particularly high because the fibers have a relatively high positive radial CTE.

The axial thermal conductivities of composites reinforced with glass, aramid, boron, and a number of the carbon fibers are relatively low. In fact, E-glass and aramid PMCs are often used as thermal insulators. As Table 13 shows, most PMCs have relatively high thermal resistivities in the transverse direction, as a result of the low thermal conductivities of the matrix and the fibers in the radial direction. Through-thickness conductivities of laminates tend to be similar to the transverse thermal conductivities of unidirectional composites.

Table 14 shows the inplane thermal conductivities and CTEs of quasi-isotropic laminates made from the same material as in Table 13. Here again, a fiber volume fraction of 60% is assumed.

We observe that the CTEs of the quasi-isotropic composites are higher than the axial values of corresponding unidirectional composites. Note, however, that the CTEs of quasi-isotropic composites reinforced with aramid and carbon fibers are very small. By appropriate selection of fiber, matrix, and fiber volume fraction, it is possible to obtain quasi-isotropic materials with CTEs very close to zero. Note that through-thickness CTEs for these laminates typically will be positive and relatively large. However, this is not a significant issue for many applications.

Turning to thermal conductivity, we find that quasi-isotropic laminates reinforced with UHM pitch carbon fibers have an inplane thermal conductivity similar to that of aluminum alloys, while UHK pitch carbon fibers provide laminates with a conductivity more than 50% higher. Both materials have densities 35% lower than aluminum.

As mentioned above, through-thickness thermal conductivities of laminates tend to be similar to the transverse thermal conductivities of unidirectional composites, which are relatively low. However, if laminate thickness is small, this may not be a significant limitation.

Physical Properties of Metal Matrix Composites

In this section, we consider physical properties of selected unidirectional fiber-reinforced MMCs and of silicon carbide particle-reinforced aluminum MMCs.

Table 13 Physical Properties of Selected Unidirectional Polymer Matrix Composites

Fiber	Density [g/cm^3 (Pci)]	Axial CTE [10^{-6}/K (10^{-6}/°F)]	Transverse CTE [10^{-6}/K (10^{-6}/°F)]	Axial Thermal Conductivity [W/m · K (Btu/h · ft · °F)]	Transverse Thermal Conductivity [W/m · K (Btu/h · ft · °F)]
E-glass	2.1 (0.075)	6.3 (3.5)	22 (12)	1.2 (0.7)	0.6 (0.3)
Aramid	1.38 (0.050)	−4.0 (−2.2)	58 (32)	1.7 (1.0)	0.1 (0.08)
Boron	2.0 (0.073)	4.5 (2.5)	23 (13)	2.2 (1.3)	0.7 (0.4)
SM carbon (PAN)	1.58 (0.057)	0.9 (0.5)	27 (15)	5 (3)	0.5 (0.3)
UHS carbon (PAN)	1.61 (0.058)	0.5 (0.3)	27 (15)	10 (6)	0.5 (0.3)
UHM carbon (PAN)	1.66 (0.060)	−0.9 (−0.5)	40 (22)	45 (26)	0.5 (0.3)
UHM carbon (pitch)	1.80 (0.065)	−1.1 (−0.6)	27 (15)	380 (220)	10 (6)
UHK carbon (pitch)	1.80 (0.065)	−1.1 (−0.6)	27 (15)	660 (380)	10 (6)

Table 14 Physical Properties of Selected Quasi-Isotropic Polymer Matrix Composites

Fiber	Density [g/cm^3 (Pci)]	Axial CTE [10^{-6}/K (10^{-6}/°F)]	Transverse CTE [10^{-6}/K (10^{-6}/°F)]	Axial Thermal Conductivity [W/m · K (Btu/h · ft · °F)]	Transverse Thermal Conductivity [W/m · K (Btu/h · ft · °F)]
E-glass	2.1 (0.075)	10 (5.6)	10 (5.6)	0.9 (0.5)	0.9 (0.5)
Aramid	1.38 (0.050)	1.4 (0.8)	1.4 (0.8)	0.9 (0.5)	0.9 (0.5)
Boron	2.0 (0.073)	6.5 (3.6)	6.5 (3.6)	1.4 (0.8)	1.4 (0.8)
SM carbon (PAN)	1.58 (0.057)	3.1 (1.7)	3.1 (1.7)	2.8 (1.6)	2.8 (1.6)
UHS carbon (PAN)	1.61 (0.058)	2.3 (1.3)	2.3 (1.3)	6 (3)	6 (3)
UHM carbon (PAN)	1.66 (0.060)	0.4 (0.2)	0.4 (0.2)	23 (13)	23 (13)
UHM carbon (pitch)	1.80 (0.065)	−0.4 (−0.2)	−0.4 (−0.2)	195 (113)	195 (113)
UHK carbon (pitch)	1.80 (0.065)	−0.4 (−0.2)	−0.4 (−0.2)	335 (195)	335 (195)

Physical Properties of Continuous Fiber-Reinforced Metal Matrix Composites. Table 11 presents physical properties of unidirectional composites consisting of UHK pitch carbon fibers in aluminum and copper matrices. These materials both have very low, slightly negative axial CTEs for the assumed fiber volume fraction of 50%. As the table shows, the axial thermal conductivities for MMCs with aluminum and copper matrices are substantially greater than that of monolithic copper. A major advantage of having thermally conductive matrix materials is that the resulting composite transverse and through-thickness thermal conductivities are more than an order of magnitude higher than those of an epoxy–matrix composite.

Table 12 presents the properties of quasi-isotropic composites composed of aluminum and copper matrices reinforced with UHK pitch carbon fibers. Here, the fiber volume fraction of about 26% has been chosen to achieve an inplane similar to that of aluminum oxide, 6.5 ppm/K (3.6 ppm/°F). The inplane thermal conductivities of the aluminum- and copper-matrix composites are 290 W/m · K (168 BTU/h · ft · °F) and 400 W/m · K (230 BTU/h · ft · °F), respectively. These values are considerably greater than those of any other material with a similar CTE, with the exception of diamond particle-reinforced copper, which is discussed later. Because of the lower fiber volume fractions, the through-thickness thermal conductivities of these composites will be somewhat higher than those of the unidirectional composites presented in Table 11.

Physical Properties of Particle-Reinforced Metal Matrix Composites. In this section, we consider the physical properties of silicon carbide particle-reinforced aluminum and diamond particle-reinforced copper.

Physical Properties of Silicon Carbide Particle-Reinforced Metal Matrix Composites. The physical properties of particle-reinforced composites tend to be isotropic (in three dimensions). As for all composites, the physical properties of silicon carbide particle-reinforced aluminum depend on constituent properties and reinforcement volume fraction. Figure 4 shows how the CTE of (SiC)p/Al varies with particle volume fraction for typical commercial materials. Table 15 presents density and CTE for several specific volume fractions, along with data for monolithic aluminum, titanium, and steel.

Thermal conductivity depends strongly on the corresponding properties of the matrix, reinforcement, and particle volume fraction. The thermal conductivity of very pure silicon carbide is slightly higher than that of copper. However, those of commercial particles are much lower. The thermal conductivities of silicon carbide particle-reinforced aluminum used in electronic packaging applications tend to be in the range of monolithic aluminum alloys, about 160–218 W/m · K (92–126 Btu/h · ft · °F).

Physical Properties of Diamond Particle-Reinforced Copper Metal Matrix Composites. Table 12 presents the physical properties of diamond particle-reinforced copper composites, which are developmental materials. As for other particle-reinforced composites, the properties can be expected to be relatively isotropic. This material has a thermal conductivity somewhat higher than that of monolithic copper, a much lower density, and a CTE in the range of semiconductors and ceramic substrates. This unique combination of properties makes this composite an attractive candidate for electronic packaging applications.

Table 15 Physical Properties of Silicon Carbide Particle-Reinforced Aluminum

Property	Aluminum (6061-T6)	Titanium (6Al–4V)	Steel (4340)	Composite Particle Volume Fraction		
				25	55	70
CTE, 10^{-6}/K (10^{-6}/°F)	23 (13)	9.5 (5.3)	12 (6.6)	16.4 (9.1)	10.4 (5.8)	6.2 (3.4)
Thermal Conductivity, W/m · K (Btu/h · ft · °F)	218 (126)	16 (9.5)	17 (9.4)	160–220 (92–126)	160–220 (92–126)	160–220 (92–126)
Density, g/cm^3 (Pci)	2.77 (0.10)	4.43 (0.16)	7.76 (0.28)	2.88 (0.104)	2.96 (0.107)	3.00 (0.108)

Physical Properties of Ceramic Matrix Composites

As discussed in Section 1, there are many CMCs and they are at various stages of development. One of the more mature systems is silicon carbide fiber-reinforced silicon carbide (SiC/SiC). For a fabric-reinforced composite with a fiber volume fraction of 40%, the density is 2.5 g/cm^3 (0.090 Pci), the CTE is 3 ppm/K (1.7 ppm/°F), the inplane thermal conductivity is 19 W/m · K (11 Btu/h · ft · °F), and the through-thickness value is 9.5 W/m · K (5.5 Btu/h · ft · °F).

Physical Properties of Carbon/Carbon Composites

The CTE of CCCs depends on fiber type, volume fraction, and geometry and matrix characteristics. In the fiber direction, CTE tends to be negative with a small absolute value. Perpendicular to the fiber direction, composite CTE is dominated by matrix properties. As a rule, the magnitude of transverse CTE is small. Both positive and negative values have been reported.

It is well known that in some forms carbon has exceptionally high thermal conductivities. For example, pyrolitic graphite can have a thermal conductivity as high as 2000 W/m · K (1160 Btu/h · ft · °F), five times that of copper. The conductivity of some types of diamond is much higher. Some CCCs also have very high thermal conductivities. Values have been reported as high as 400 W/m · K (230 Btu/h · ft · °F) for quasi-isotropic composites and 700 W/m · K (400 Btu/h · ft · °F) for unidirectional materials.

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CHAPTER 13

SMART MATERIALS

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1 INTRODUCTION

The world has recently undergone two materials ages, the plastics age and the composite age. In the midst of these two ages a new era has developed. This is the smart materials era. According to early definitions, smart materials are materials that respond to their environments in a timely manner.[1–4]

The definition of smart materials has been expanded to materials that receive, transmit, or process a stimulus and respond by producing a useful effect that may include a signal that the materials are acting upon it. Some of the stimuli that may act upon these materials are strain, stress, temperature, chemicals (including pH stimuli), electric field, magnetic field, hydrostatic pressure, different types of radiation, and other forms of stimuli.[5]

The effect can be caused by an absorption of a proton, of a chemical reaction, of an integration of a series of events, of a translation or rotation of segments

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Handbook of Materials Selection. Edited by Myer Kutz

within the molecular structure, of a creation and motion of crystallographic defects or other localized conformations, of an alteration of localized stress and strain fields, and of others. The effects produced can be a color change, a change in index of refraction, a change in the distribution of stresses and strains, or a volume change.[5]

Another important element of a smart material is that the action of receiving stimuli and responding to the stimuli to produce a useful effect is that it must be reversible. Another feature that is an important factor in determining if a material is smart pertains to its asymmetrical nature. This is primarily critical for the piezoelectric materials. Other types of smart materials exhibit this trait. However, little research has been performed to verify this observation.

Also, it should be pointed out that the word *intelligent* is used to describe smart materials. The notation *smart* has been overused as a means to market materials and products.

From the purist point of view materials are smart if at some point within their performance history they act reversible to a stimulus. Materials that formally have the label of being smart include piezoelectric materials, electrostrictive materials, electrorheological materials, magnetorheological materials, thermoresponsive materials, pH-sensitive materials, ultraviolet-(UV) sensitive materials, smart polymers, smart gels (hydrogels), smart catalysts, and shape memory alloys. In this treatment of the subject we will be using some of these classifications and in some cases, the classification of a particular material may appear to be in error. This will be done to illustrate the rapid growth of the field of smart materials and the rediscovery of the smart behavior of materials known for centuries. As we continue to better understand the smartness of materials, our definitions will change. In each material section there will be discussions pertaining to the material definition, types of materials that belong to that class, properties of the members, and applications of the materials. In some cases a more detailed discussion of application will be given to both illustrate the benefit of these materials and to simulate the use of these materials in new applications. This is being done in the spirit of this handbook.

Another important feature of smart materials is their inclusion in structures. These are known as smart structures. Smart structures are simply structures with at least one smart material incorporated within its structure and that from the effect of the smart material cause an action. A smart structure may have sensors (nerves), actuators (muscles), and a control (brain). Thus, you can see the term *biomimetric* associated with smart structures. Smart structures are being designed to make our life more productive and easy. And with the number of sensors, actuators, and control systems available, coupled with the materials and the genius of scientists and engineers, these structures are being more noticeable and numerous. Reading nontechnical magazines, watching television, and going to stores can verify this.

In a silly way the growth of smart structures can be illustrated by an article that appeared on page D11 of the Thursday 14, 2000, issue of the *New York Times*. This article describes the "Big Mouth Billy Bass," the singing fish. The article described the smartness of this novel toy.

Examples of technical applications of smart structures are structures such as composite materials embedded with fiber optics, actuators, sensors, microelectromechanical systems (MEMS), vibration control, sound control, shape control, product health or lifetime monitoring, cure monitoring, intelligent processing, active and passive controls, self-repair (healing), artificial organs, novel indicating devices, designed magnets, damping, aeroelastic stability, and stress distributions. Smart structures are found in automobiles, space systems, fixed- and rotary-wing aircrafts, naval vessels, civil structures, machine tools, recreation, and medical devices.[5,6]

Another feature that is important about smart materials and structures is that they encompass all fields of science and engineering. Thus when searching for information on smart materials and structures, there are numerous sources, web sites, and professional societies that deal with the technology.

2 PIEZOELECTRIC MATERIALS

The simplest definition of piezoelectric materials can be obtained by first breaking the word into *piezo* and *electric*. *Piezo* is from the Greek word *piezein* that means to press tightly or squeeze. Combining *piezein* with electric we have ‘‘squeeze electricity.’’ And that to the first estimation is a very good definition.

The history of piezoelectric materials is relatively simple and only the highlights will be presented. In 1880, Pierre and Paul-Jean Curie showed the piezoelectric effect in quartz and Rochelle salt crystals. Their first observations were made by placing weights on the faces of particular crystal cuts, like the X cut quartz plate, detecting charges on the crystal surfaces, and demonstrating that the magnitude of charge was proportional to the applied weight. This phenomenon has become to be known as the direct pressure electric piezoelectric effect. In 1881 G. Lippmann made the prediction that a crystal such as quartz would develop a mechanical strain when an electric field was applied. In the same year the Curies reported the converse pressure electric piezoelectric effect with quartz and Rochelle salt. They showed that if certain crystals were subjected to mechanical strain, they became electrically polarized and the degree of polarization was proportional to the applied strain. The French inventor Langevin developed the first SONAR, using quartz crystals in 1920. During the 1940s researchers discovered and developed the first polycrystalline, piezoelectric ceramic, barium titanate. One of the significant advantages of piezoelectric ceramics over piezoelectric crystals is their ability to be formed in a variety of shapes and sizes. In 1960, researchers discovered a weak piezoelectric effect in whalebone and tendon that lead to intense search for other piezoelectric organic materials. And in 1969, Kawai found very high piezoelectric activity in polarized polyvinylidene fluoride.[7,8]

The piezoelectric effect exists in a number of naturally occurring crystals such as quartz, tourmaline, and sodium potassium tartrate. For a crystal to exhibit the piezoelectric effect, it must not have a center of symmetry. When a stress (tensile or compressive) is applied to a crystal with a center of symmetry, it will alter the spacing between the positive and negative sites in each elementary cell unit, thus causing a net polarization at the crystal surface. The effect is approximately linear. The polarization is directly related to the applied stress. It is directional dependent. Thus, compressive and tensile forces will generate electric fields and voltages of opposite polarity. The effect is also reciprocal; thus when the crystal is exposed to an electric field, it undergoes an elastic strain changing its length based upon the field polarity.

As previously mentioned, the ceramic-type materials play an important role in the area of piezoelectrics. Piezoelectric ceramics are polycrystalline ferroelectric materials with a perovskite crystal-type structure. The crystal structure is tetragonal/rhombahedral with a close proximity to cubic in nature. Piezoelectric ceramics have the general formula of $A^{2+}B^{4+}O_3^{2-}$. The letter A represents a large divalent metal ion such as barium or lead, and B is one or more tetravalent metal ion such as titanium or zirconium or manganese. These ceramics are considered to be masses of minute crystallities that change crystal forms at the Curie temperature. Above the Curie temperature, the ceramic crystallities have a simple cubic symmetry. This form is centrosymmetric with positive and negative charge sites coinciding, thus there are no dipoles present. The material is considered to be paraelectric. Below the Curie temperature, the ceramic crystallities have a tetragonal symmetry; this form lacks a center of symmetry with the positive and negative charge sites that no longer coincide, thus each unit cell now has an electric dipole that may be reversed and switched to certain direction by the application of an electric field. The material is now considered to be ferroelectric.

The piezoelectric properties of the ceramics can be enhanced by applying a large electric field at an elevated temperature, thus generating an internal remnant polarization that continues long after the removal of the electric and thermal fields. This technique is known as poling. The poling of piezoelectric ceramics has eliminated the use of piezoelectric crystals in many applications.

The vinylidene fluoride monomer, $CH_2{=}CF_2$, yields the semicrystalline polymer, polyvinylidene fluoride (PVDF), upon polymerization. This polymer was found to be highly piezoelectric. Polyvinylidene fluoride is manufactured in sheet form from the nonpolar α-phase film extruded from the melt. The extruded film is uniaxially stretched. This process rotates the long polymer chains and forms the polar beta phase. The beta phase is needed for the high piezoelectricity. The final step consists of reorientation of the randomly directed dipoles associated with the stretched β phase by applying a poling field in the direction normal to the plane of the film. The resulting piezoelectric polyvinylidene fluoride has orthotropic symmetry.

Copolymers and rubber and ceramic blends of polyvinylidene fluoride have been prepared for used as piezoelectric materials. The most common copolymer is one based on the polymerization reaction of vinylidene fluoride and trifluoroethylene. Polyvinylidene fluoride and its copolymers with trifluoroethylene have low mechanical quality factors, high hydrostatic mode responses, and their acoustic impedance is similar to water, thus making them ideal for underwater hydrophone applications.[9]

A series of polyimides has been developed at NASA Langley for use in piezoelectric applications. These polyimides have pendant trifluoromethyl (—CF3) and cyano (—CN) polar groups. Whenever these polyimides are exposed to applied voltage of the order of 100 MV/m at elevated temperatures, the polar groups developed a high degree of orientation thus resulting with polymer films with high piezoelectric and pyroelectric properties. The piezoelectric response for these polyimides is in the same vicinity to those of polyvinylidene fluoride at room temperature. However, the piezoelectric response of the polyimides is greater at elevated temperatures.[10]

Multiphase piezoelectric composites have been developed for their synergetic effect between the piezoelectric activity of monolithic ceramics and the low density of nonpiezoelectric polymeric materials. One class of piezoelectric composites that has been developed is the smart tagged composites. These piezoelectric composites are PZT-5A particles embedded into an unsaturated polyester polymer matrix and are used for structural health monitoring. Conductive metal-filled particles with polyimides films have been explored for microelectronic applications. The use of graphite-filled polyimides has shown potential as microsensors and actuators.

Piezoelectric materials have been used in thousands of applications in a wide variety of products in the consumer, industrial, medical, aerospace, and military sectors. When a piezoelectric material is subjected to a mechanical stress an electric charge is generated across the material. The ability of a material to generate a charge or electric field when subjected to a stress is measured by the piezoelectric voltage coefficient (g).

The converse effect occurs when a piezoelectric material becomes strained when placed in an electric field. Under constant stress conditions, the general equation for the piezoelectric charge coefficient (d) can be expressed as the change in the strain (S) of the piezoelectric material as a function of the applied electric field (E).

In should be denoted that piezoelectric materials are also pyroelectric. They generate electric charge as they undergo a temperature change. Whenever their temperature is increased, a voltage develops having the same orientation as the polarization voltage. Whenever the temperature is decreased, a voltage develops with an opposite orientation opposite to the polarization voltage.[11]

Tables 1–3 show some of the applications of materials that utilize direct and converse piezoelectric effects and the applications of polyvinylidene fluoride piezoelectric film. Specific applications will be discussed.

One of the uses of piezoelectric ceramics and polymers is their application in ink-on-demand printing. Several commercially available ink jet printers are

Table 1 Applications of Piezoelectric Materials Utilizing the Converse Effect[8]

Process	Application
Load cells	Force measurements Pressure measurements
Velocity sensors	Velocity measurements
Accelerometer	Acceleration measurements Vibration monitoring
Structural monitoring	Detection of acoustic emissions
Hydrophones	Monitoring of marine life Monitoring of the heart Monitoring of circulatory systems
Igniters	Gas ignition in welders, barbeques, lighters
Remote control	Sensor in commercial remote controls
Microphones	Detection of audible frequencies

Table 2 Applications of Piezoelectric Materials Utilizing the Converse Effect[8]

Process	Application
Welding	Welding rigid thermoplastics
	Seam welding film and fabric
	Metal-in-plastic insertion
	Metal microbonding
	Lap welding of high electrical conductivity and dissimilar materials
	Seam welding sheets
Machining	Prophylaxis teeth dental treatment
	Vibration-assisted rotary machining of hard, brittle materials
	Impact grinding with abrasive slurries
	Vibration-assisted drilling, tapping, and turning
Forming	Drawing thin-wall metal tubing of large diameter-to-wall ratios
	Drawing small-diameter wire from difficult to form metals
Cutting	Vibration-assisted cutting of fibrous and spongy materials
Cleavage	Cleaving crystals and laminated objects
Densification	Compaction of powder

Table 3 Applications of Polyvinylidene Fluoride Piezoelectric Film[7]

- Medical
  - Diagnostics: Apnea monitor, blood pressure cuff, pulse counter, stethoscope, sleep disorder monitor, respiratory airflow, patient bed monitor
  - Ultrasound: near-field imaging, prostrate, transdermal, transluminal, coronary arterial, breast, lithotripter, hydrophone calibration
  - Handicapped aides: switches, braille reader, hearing aid, speech intensification
  - Implantables: pacemaker activity monitor, vasecular graft monitor, micropower source
  - Instrumentation: intravenous drop counter, IV air bubble detector, laser switch/monitor
- Automotive: accelerometers, occupancy seat sensor, compartment switches, horn, fuel level, tire rotation, security, keyless entry, motion (theft) sensor
- Consumer
  - Musical instruments: pick-up, drum trigger
  - Sports equipment: target location, reaction time, foul time, force, sweet spot
  - Toys/games: switches, proximity (air ranging, pyro), novelty speakers, target scoring
  - Audio: tweeter, balloon speaker, novelty speaker (visor, poster), microphone, speaker distortion feedback, electronic piano keys
  - Appliance: washer imbalance, vacuum soil sensing, dishwasher
- Computer input/output: keypads, digitizers, mouse, joystick, pen, ink jet droplet generator, ink jet droplet detector, toner and ink jet level, coin counting, disk drive shock sensing
- Industrial
  - Switches: solid state, snap action, cantilever beam, keypad, vandal proof
  - Robotics: tactical sensor, micropositioner, safety mats and switches, bumper impact
  - Security and energy management: glass break detection, floor/mat sensors, penetration detection, flame detection
  - Flow/level: fan monitor, fluidic oscillator, doppler ultrasound, solid-state fluid level, laminar/turbulent layer
- Instrumentation
  - Machine health monitor: accelerometer, contact microphone, strain gauging
  - Weather sensors: rain intensity, hail detection, wind velocity
  - Active vibration damping: strain gauge array, actuator array
  - Nondestructive testing: acoustic emission
  - Ranging: distance, safety
  - Adaptive optics: fiber optic shutter, deformable mirror, laser scanner
  - Oil exploration: hydrophone, seismic geophone

based upon this technology.[12,13] In this application the impulse ink jet is produced using a cylindrical transducer that is tightly bound to the outer surface of a cylindrical glass nozzle with an orifice ranging from mils to microns in diameter. In addition to this application several industrious researchers have expanded upon this technique by using a printer as a chemical delivery system for the application of doped polymers for organic light-emitting displays. A group of researchers from Princeton University has used a color ink jet printer based upon piezoelectric technology with a resolution of 640 dots per line and simply replaced the inks with polymer solutions.[14] This technique has also been applied to the manufacture of color filters for liquid-crystal displays.

Another application of the use of piezoelectric polymer film involves the work of a group of researchers at the Thiokol Company and an earlier work[15,16] to monitor adhesive joints. The study showed that during the bonding process, the polyvinylidene fluoride piezoelectric film sensors monitored the adhesive cure ultrasonically and qualitatively determined the presence of significant void content. The study also indicated that normal bond stresses were quantified during cyclic loading of single lap joints and electrometric butt joints. The researchers revealed that this was a significant step toward service life predictions. But, more and better-understood monitoring techniques are needed.

An interested application of piezoelectric ceramics can be illustrated in the hundred of vertical pinball machines popular in the Pachinko parlors of Japan. The machines are assembled with stacks of piezoelectric disks that can act as both sensors and actuators. When a ball falls on the stack, the force of impact generates a piezoelectric voltage pulse that in turns generates a response from the actuator stack through a feedback control system. The stack expands, thus throwing the ball out of the hole and moving up a spiral ramp through a sequence of events, and finally repeating the sequence.[17]

3 ELECTROSTRICTIVE MATERIALS

Piezoelectric materials are materials that exhibit a linear relationship between electric and mechanical variables. Piezoelectricity is a third-rank tensor. Electrostrictive materials also show a relationship between these two variables. However, in this case, it is a quadratic relationship between mechanical stress and the square of electrical polarization. Electrostriction can occur in any material and is a small effect. One difference between piezoelectric and electrostrictive materials is the ability of the electrostrictive materials to show a larger effect in the vicinity of its Curie temperature. Elecrostriction is a fourth-rank tensor property observed both in centric and acentric insulators. This is especially true for ferroelectric materials such as the members of the perovskite family. Ferroelectrics are ferroic solids whose domain walls have the capability of moving by external forces or fields. In addition to ferroelectrics the other principal examples of ferroic solids are ferromagnetics and ferroelastics, both of which have potential as smart materials. Other examples of electrostrictive materials include lead manganese niobate: lead titanate (PMN: PT) and lead lanthanium zirconate titanate (PLZT).

An interesting application of electrostrictive materials is in active optical applications. During the Cold War, the satellites that flew over the Soviet Union used active optic systems to eliminate the atmospheric turbulence effects. Electrostrictive materials have the advantage over piezoelectric materials in adjusting the position of optical components due to the less amount of hysteresis associated with the motion. Work on active optic systems has continued. Similar multilayer actuators were used to correct the position of the optical elements in the Hubble telescope. Supermarket scanners use actuators and flexible mirrors to read bar codes optically.[17]

Other examples of this family may be included in the electroactive polymers. This name has appeared in the technical literature for approximately a decade with an increasing interest in the last couple of years. Electroactive polymers include any polymer that is simulated by electricity and responds to its effect in a reversible manner. This classification is sort of a melting pot of smart materials.[18]

4 MAGNETOSTRICTIVE MATERIALS

Magnetorestrictive materials have the material response of mechanical deformation when stimulated by a magnetic field. Shape changes are the largest in ferromagnetic and ferrimagnetic materials. The repositioning of domain walls that occur when these solids are placed in a magnetic field leads to hysteresis between magnetization and an applied magnetic field. When a ferromagnetic material is heated above its Curie temperature, these effects disappear. The microscopic properties of a ferromagnetic solid are different than a ferrimagnetic solid. The magnetic dipoles of a ferromagnetic solid are aligned parallel. The alignment of dipoles in a ferrimagnetic solid can be either parallel or in other directions.[5,19,20]

Magnetorestrictive materials are usually inorganic in chemical composition and alloys of iron, nickel, and doped with rare earths. The most effective magnetorestrictive material is another alloy developed at the Naval Ordinance Laboratory, TERFENOL-D. It is an alloy of terbium, dysprosium, and iron with a formula of $Tb_xDy_{1-x}Fe$ with x between 0.27 and 0.30 and y between 1.90 and 1.98. The full effect of magnetorestriction occurs in crystalline materials. One of the hindrances preventing magnetorestrictive materials reaching commercial significance has been cost. Over the past three decades there has been a great deal of development of giant magnetorestrictive materials, colossal magnetorestrictive materials, and organic and organometallic magnets.[5,21]

The giant magnetorestrictive effect was first observed in iron-chromium laminates in 1988. These laminates consisted of alternating layers of 50-Å-thick iron with chromium layers of various thicknesses. The iron layers oriented themselves with antiparallel magnetic moments. A magnetic field of 20 kOe applied in the plane of the iron layers will uncouple this antiferromagnetic orientation. Since the magnetic orientation of one layer is controlled, another layer is free to rotate with an applied field; this change in orientation of the two layers produces a magnetorestrictive effect. This discovery of giant magnetorestrictive materials enhanced the increased sensitivities in the hard disk drive heads at a cost-effective price.

In the 1990s, researchers further enhanced the field of magnetorestrictive materials with the development of colossal magnetorestrictive materials. Ratios of magnetorestrictive effect in excess of 100,000% were observed. These new materials were found to be epitaxial lanthanum–calcium–manganese–oxygen thin films, polycrystalline lanthanum–yttrium–calcium–manganese–oxygen and polycrystalline lanthanum–barium–manganese–oxygen. These again enhanced the product performance at costs less that the original magnetorestrictive materials.[21]

Magnets that exhibit commercial potential should have magnetic saturation and coercive field properties that are operational at low temperatures. Magnets are useful below their critical temperature. Most inorganic magnets have critical temperatures well above room temperature.

The first organometallic magnet was the ionic salt complex of ferric bis(pentamethylcyclopentadienide) and tetracyanoethylene. This complex was a ferromagnet below its critical temperature of 4.8 K. The highest effective critical temperature of an organometallic magnet was in the vicinity of 400 K.[22]

Organic magnets are different than organometallic magnets for two reasons. The first is obvious. Organic-based magnets do not contain metal atoms. This causes a great deal of rethinking the principles of magnetism. The second difference refers to the fact the coupled spins of the organic magnets reside only in the p orbitals while the coupled spins of the organometallic magnets can reside in either the p orbitals or the d orbitals or a combination of the two. A series of organic magnets based upon the nitroxide chemistry was synthesized and their magnetic behavior studied between 0.6 and 1.48 K. The most studied compound of these nitroxide-based magnets was 4-nitrophenylnitronyl nitroxide. This compound showed a saturation magnetization equivalent to one spin per molecule. This is indicative of ferromagnetic behavior.[5,22]

5 ELASTORESTRICTIVE MATERIALS

This class of smart materials is the mechanical equivalent to electrorestrictive and magnetorestrictive smart materials. These smart materials exhibit high hysteresis between stress and strain. The motion of ferroelastic domain walls causes the hysteresis. This motion of the ferroelastic domain walls is very complicated and complex near a martensitic phase transformation. At this phase change, two types of crystal structural changes occur. One is induced by mechanical stress and the other is by domain wall motion. Martensitic shape memory alloys have wide, diffuse phase changes and the ability to exist in both high- and low-temperature phases. The domain wall movements disappear with total change to the high-temperature phase.[5,19,20] The elastorestrictive smart material family is in its infancy.

6 ELECTRORHEOLOGICAL MATERIALS

The rheological materials are an exciting group of smart materials. Electrorheological and magnetorheological materials can change their rheological properties instantly through the application of either an electric or magnetic field. The electrorheological materials (fluids) have been known for several centuries. The rheological or viscous properties of these fluids, which are usually uniform dispersions, or suspensions of particles within a fluid are changed with the application of an electric field. The mechanism of how these electrorheological fluids work is simple. In an applied electric field the particles orient themselves in fiberlike structures (fibrils). When the electric field is off, the fibrils disorient themselves. Another way to imagine this behavior is to consider logs in a river.

If the logs are aligned, they flow down the river. If they are disordered, they will cause a log jam thus clogging up the river. A typical example of an electrorheological fluid is a mixture of corn starch in silicone oil. Another fluid that has been experimented with as a replacement for the silicone oil has been chocolate syrup. Another feature of electrorheological systems is that their damping characteristics can be changed from flexible to rigid and vice versa. Electrorheological fluids were evaluated using a single-link flexible-beam test bed. The beam was a sandwich configuration with electrorheological fluids distributed along its length. When the beam was rapidly moved back and forth, the electrorheological fluid was used to provide flexibility during the transient response period of the maneuver for speed and made rigid at the end point of the maneuver for stability. A practical way of viewing this behavior is to compare it with fly fishing.[5] It has been suggested to use rheological fluids in the construction of fishing rods and golf clubs.

7 MAGNETORHEOLOGICAL MATERIALS

The magnetorheological materials (fluids) are the magnetic equivalent of electrorheological fluids. These fluids consist of either ferromagnetic or ferrimagnetic particles that are either dispersed or suspended within; and the applied stimulus is a magnetic field. A simple magnetorheological fluid consists of iron powder in motor oil. The Lord Corporation provided a clever demonstration of magnetorheological fluids. They supplied an interlocking two-plastic-syringe system filled with a magnetorheological fluid and two small magnets. The fluid flows freely, without the magnets placed in the middle of the two syringes. With the two magnets in place, the fluid flow completely stops.

An interesting adaptation of magnetorestrictive fluids is a series of elastomeric matrix composites embedded with iron particles. During the thermal cure of the elastomer, a strong magnetic field was applied to align the iron particles into chains. These chains of iron particles were locked into place within the composite through the cross-linked network of the cured elastomer. If a compressive force stimulated the composite, it was 60% more resistant to deformation in a magnetic field. If the composite was subjected to a shear force, its magnetic field induced modulus was an order of magnitude higher than its modulus in a zero magnetic field.[5,23]

A recent well-thought article described attempts to enhance the properties of epoxies with magnetic fields. The study did show the low conversion rates of the epoxy with the hardener; economically generated magnetic fields had an effect on the properties of the final composite. At the high conversion of the reactants there exists a need to drive the scarce unreacted glycidyl and amine functionalities together. And only the magnetic fields generated by superconducting electromagnets are capable of this.[24]

There continues to be a great deal of research into magnets and magnetism. A new area of research involves magnetic nanocomposite films. Magnetic particles exhibit size effects. Below a critical size, magnetic clusters comprise single domains, whereas with bulk materials there are multiple domains. Nanomagnets show unusual properties such as superparamagnetism and quantum tunneling of magnetization. These unique properties of magnetic nanoclusters can lead to applications in information storage, color imaging, magnetic refrigeration, ferrofluids, cell storing, medical diagnosis, and controlled drug delivery. A nanocomposite is considered to be the incorporation of these nanoclusters into polymeric matrices such as polyaniline.[25]

8 THERMORESPONSIVE MATERIALS

Amorphous and semicrystalline thermoplastic polymeric materials are unique due to the presence of a glass transition temperature. At their glass transition temperatures, the specific volume of polymer and its rate of change changes. This transition affects a multitude of physical properties. Numerous types of indicating devices could be developed based upon the stimuli-response (temperature-specific volume) behavior. This chapter contains several examples of this type of behavior; however, its total impact is beyond the scope of this chapter. To take advantage of this behavior in product development or material selection it requires reviewing the necessary requirements in the many polymer references.

A few examples that illustrate thermoresponsive behavior that are a little unique are being included in this chapter. One refers to the polyethylene/poly(ethylene glycol) copolymers that were used to functionalize the surfaces of polyethylene films.[5,26] One may refer to this illustration as being a ‘‘smart surface’’ or functionally gradient surfaces. When the film is immersed in an aqueous dispersion of the copolymer, the ethylene glycol moieties attach to the polymer film surface resulting in a film surface having solvation behavior similar to poly(ethylene glycol) itself. Due to the inverse temperature-dependent solubility behavior of poly(alkene oxide)s in water surface modified polymers are produced that reversibly change their hydrophilicity and solvation with changes in temperatures.[27] Similar behaviors have been observed as a function of changes in pH.[28–31]

Another interesting example of materials that respond smartly to changes in temperature are the fabrics based upon poly(ethylene glycol)-modified cottons, -modified polyesters, and -modified polyamide/polyurethanes. A combination of the thermoresponsiveness of these fabrics with a sensitivity to moisture resulted in a family of fabrics that can serve as smart pressure bandages. When exposed to an aqueous medium such as blood, these fabrics contract and apply pressure. Once the fabric dries, it releases the pressure.[23]

Polymers based upon the monomer vinyl methyl ether have the unique behavior of shrinking upon heating to approximately 40°C. In the right design with normal behaving polymers, one can construct a device that can grasp objects like a hand.

9 pH-SENSITIVE MATERIALS

By far the widely known chemical classes of pH-sensitive materials are the acids, bases, and indicators. The indicators fit the definition of smart materials by changing colors, as a function of pH and the action is reversible.

Other examples of pH-sensitive materials include some of the smart gels and smart polymers mentioned in this chapter. There are a large number of pH-sensitive polymers and gels that are used in biotechnology and medicine. Usually these materials are prepared from various combinations of such monomers and polymers such as methacrylic acid, methyl methacrylate, carboxymethyethylcellulose, cellulose acetate, cellulose phthalate, hydroxypropylmethylcellulose phthalate, hydroxypropylmethylcellulose acetate, hydroxypropylmethylcellulose succinate, diethylaminoethyl methacrylate, and butyl methacrylate.[32]

10 LIGHT-SENSITIVE MATERIALS

There are several different material families that exhibit different types of behavior to a light stimulus. Electrochromism is a change in color as a function of an electrical field. Other types of behavior for light-sensitive materials are thermochromism, color change with heat, photochromism, color change with light, and photostrictism, shape changes caused by changes in electronic configuration due to light.[5,19,20]

Electrochromic smart windows have been intensively researched over the past few decades. There have been over 1800 patents issued for optical switching devices with the bulk of these being issued in Japan. A typical switchable glass is multilayered with an electrochromic device embedded inside. A window device may have glass with an interior conductive oxide layer both on the top and bottom. Inside of the sandwich of glass and conductive oxide is the electrochromic device. This device consists of an electrochromic layer, an ion storage layer, and between these two layers, an ion conductor.

An interesting light-sensitive material with both electro- and thermochromism behaviors, Li_xVO_2, was evaluated for a smart window application.[33]

Materials have been developed to exhibit both photochromic and photographic (irreversible behavior to light) behaviors. One such system is based upon a substituted indolinospirobenzopyran embedded in a polystyrene matrix. This system performs as a photochromic system at low exposure in the ultraviolet range and as a photographic system at high exposures. The image can be devisualized by heat and can be restored with UV irradiation many times.[34]

11 SMART POLYMERS

The term *smart polymers* was almost dropped as a classification for smart materials in this treatment of the subject. It is very confusing. Each field of science and engineering has its own definition for a smart polymer and each can fit in another classification, and its distinction in smartness can be confusing at times. It is being included as a separate class due to several excellent articles written with smart polymers in the title.

In medicine and biotechnology, smart polymer systems usually pertain to aqueous polymer solutions, interfaces, and hydrogels. Smart gels or hydrogels will be treated separately. Smart polymers refer to polymeric systems that are capable of responding strongly to slight changes in the external medium: a first-order transition, accompanied by a sharp decrease in the specific volume of the polymer. If the external medium is temperature, this transition is known as the glass transition temperature of the polymer and several properties of the polymer changes. These properties include volume, coefficient of thermal expansion, specific heat, heat conductivity, modulus, and permeation. Manipulating the detection around the glass transition temperature of the polymer can develop smart devices. There are numerous examples in product development that have resulted in failure because of not taking into account the glass transition temperature of the polymer. And it should be noted, as the polymer cools down from high temperatures and goes below its glass transition temperature, its below glass transition properties are returned and vice versa.

Smart polymers can respond to stimuli such as temperature, pH, chemical species, light, UV radiation, recognition, electric fields, magnetic fields, and other types of stimuli. The resulting response can be changes in phases, shape, optics, mechanical strengths, electrical and thermal properties, reaction rates, and permeation rates.

12 SMART (INTELLIGENT) GELS (HYDROGELS)

In the literature you can find these smart materials under a variety of names as reflected by the title of this section. The concept of smart gels is a combination of the simple concept of solvent-swollen polymer networks in conjunction with the material being able to response to other types of stimuli. A partial list of these stimuli includes temperature, pH, chemicals, concentrations of solvents, ionic strengths, pressure, stress, light intensity, electric fields, magnetic fields, and different types of radiation.[35–39] The founding father of these smart gels, Toyochi Tanaka, first observed this phenomenon in swollen clear polyacrylamide gels. Upon cooling, these gels would cloud up and become opaque. Upon warming, these gels regained their clarity. Upon further investigation to explain this behavior, it was found that some gel systems could expand to hundreds of times their original volume or could collapse to expel up to 90% of their fluid content with a stimulus of only a 1°C change in temperature. Similar behavior was observed with a change of 0.1 pH units.

These types of behaviors lead to the development of gel-based actuators, values, sensors, controlled-release systems for drugs and other substances, artificial muscles for robotic devices, chemical memories, optical shutters, molecular separation systems, and toys. Other potential systems for the development of products with smart (intelligent) gels (hydrogels) include paints, adhesives, recyclable absorbents, bioreactors, bioassay systems, and display.

There are numerous examples of the commercialization of these smart gels in one of the cited reference.[35] This chapter will only include a few examples of smart gels. One such smart gel consists of an entangled network of two polymers, a poly(acrylic acid) (PAA) and a triblock copolymer of poly(propylene oxide) (PPO) and poly(ethylene oxide) (PEO) with a sequence of PEO–PPO–PEO. The PAA portion is a bioadhesive and is pH responsive, the PPO moieties are hydrophobic that assist solubilize lipophilic substances in medical applications, and the PEO functionalities tend to aggregate thus resulting in gelation at body temperatures. Another smart gel system with a fairly complex composition consists of citosan, a hydrolyzed derivative of chitin (a polymer of *N*-acetylglucosamine that is found in shrimp and crab shells), a copolymer of poly(*N*-isopropylacrylamide) and poly(acrylic acid), and a graft copolymer of poly(methacrylic acid) and poly(ethylene glycol). This gel system was developed for the controlled release of insulin in diabetics. Polyampholytic smart hydrogels swell to their maximum extent at neutral pH values. When such gels, copolymers of methacrylic acid 2-(*N*,*N*-dimethylamino)ethyl methacrylate, are subjected to either acidic or basic media, they undergo rapid dehydration.[39]

One very unusual smart gel is based upon the polymerization of *N*-isopropylacrylamide, a derivative of tris(2,2′-bipyridyl)ruthenium(II) that has a polymerizable vinyl group, and *N*,*N*′-methylenebisacrylamide. It is a self-oscillating gel that simulates the beating of the heart with color changes.[40]

13 SMART CATALYSTS

The development of smart catalysts is a new field of investigation and has shown a great deal of activity in the universities of the oil-producing states. One such smart catalyst is rhodium-based with a poly (ethylene oxide) backbone. Smart catalysts such as this one function opposite as a traditional catalyst, that is, as the temperature increases, it becomes less soluble, precipitating out of the reaction solution, thus becoming inactive. As the reaction solution cools down, the smart catalyst redissolves and thus becomes active again.[5,23] Other smart catalyst systems are being developed that dissociate at high temperatures (less active) and recombine at low temperatures (more active).[5,27]

14 SHAPE MEMORY ALLOYS

The shape memory effect in metals is a very interesting phenomenon. Just imagine taking a piece of metal and deforming it completely and then restoring it to its original shape with the application of heat. Taking a shape memory alloy spring and hanging a weight on one end of the spring can easily illustrate this. After the spring has been stretched, heat the spring with a hot air gun and watch it return to its original length with the weight still attached.

These materials undergo a thermomechanical change as they pass from one phase to another. The crystalline structure of such materials as nickel-titanium alloys enters into the martensitic phase as the alloy is cooled below a critical temperature. In this stage the material is easily manipulated through large strains with a little change in stress. As the temperature of the material is increased above the critical temperature, it transforms into the austentic phase. In this phase the material regains its high strength and high modulus and it behaves normally. The material shrinks during the change from the martensitic to the austentic phase.[5,19,20]

The nickel–titanium alloys have been the most used shape memory material. This family of nickel–titanium alloys is known as Nitinol (*Ni*ckel *Ti*tanium *N*aval *O*rdinance *L*aboratory), named in part for the laboratory where this material was first observed. Nitinol has been used in military, medical, safety, and robotics applications. Specific applications include hydraulic lines used on F-14 fighter planes, medical tweezers and sutures, anchors for attaching tendons to bones, stents for cardiac arteries, eyeglass frames, and antiscalding valves used in water faucets and showers.[5,41,42]

In addition to the family of nickel–titanium alloys there are other alloys that exhibit the shape memory effect. These alloys are silver–cadmium, gold–cadmium, copper–aluminum–nickel, copper–tin, copper–zinc, combinations of copper–zinc with silicon or tin or aluminum, indium–thallium, nickel–aluminum, iron–platinum, manganese–copper, and iron–manganese–silicon.[43] Not all combinations of either the two or three elements yields an alloy with the shape memory effect thus it is recommended to review the original literature.

In the open literature there are several articles from Mitsubishi Heavy Industries, Ltd. describing the room temperature functional shape memory polyurethanes. To this writer, these studies only attest to the behavior of a polymer at its glass transition temperature. Thus if you wish to describe a polymer's behavior at its glass transition temperature and since that free volume change is reversible, you may call it a smart polymer or a shape memory material or a thermoresponsive material. The unique characteristic of these polyurethanes is that their transition occurs in the vicinity of room temperature.[44,45]

15 UNUSUAL BEHAVIORS OF MATERIALS

As one researches the field of smart materials and structures, one realizes that there are many smart materials or that there are many material behaviors that are reversible within their lifetime. The ability to develop useful products from smart materials is left up to one's imagination. For example, water is a very unique material. It expands upon freezing. And as we know, the force generated by this expansion causes sidewalks and highways to crack. Now, what if you surround water pipes with a heating system that consists of a heater and water enclosed in piezoelectric polymer or elastomer container that is in a fixed space. As the temperature drops to the freezing point of water, it expands and generates force against the piezoelectric container which in turn generates electricity, thus powering the heater keeping the pipes from freezing.

As previously mentioned, sometimes it is difficult to classify the material and its behavior. One such case involves an assigned patent to the University of Illinois. The title of the patent is "Magnetic gels which change volume in response to voltage changes for magnetic resonance imaging."[46] The patent teaches the use of a matrix that has a magnetic and preferably superparamagnetic component and the capability of changing its volume in an electric field.

Fullerenes are spherically caged molecules with carbon atoms at the corner of a polyhedral structure consisting of pentagons and hexagons. The most stable of the fullerenes is a C60 structure known as buckyball or buckminsterfullerene. The fullerenes have been under commercial development for the past decade. One application of the fullerenes as a smart material consists of embedding the fullerenes into sol–gel matrices for the purpose of enhancing optical limiting properties.[47]

A semiconducting material with a magnetic ordering at 16.1 K was produced from the reaction of buckyball with tetra(dimethylamino)ethylene. This organic-based magnet had neither the coercivity nor saturation magnetization to function totally as a ferromagnet. The replacement of buckyball with higher carbon number fullerenes in the reaction with tetra(diamethylamino)ethylene did not produce any complexes that showed magnetic ordering.[22]

16 COMMENTS, CONCERNS, AND CONCLUSIONS

In dealing with smart materials and structures, there is still a lot of confusion as to the name of these materials and what makes a material or structure smart. There are numerous products with *Smart* in the name that do not meet the definition of being smart, that is, responding to its environment in a reversible manner. The scientist/engineer should not fault the advertising professional for doing his or her job whenever they use the term in a product description. But I must admit I chuckle to myself whenever I see the magazine *Smart Money*. Does this mean that after I spent money it will return to me because it is reversible?

The confusion also exists in the smart materials community with the term *smart polymers*. The applications of most smart polymers center around the polymer's glass transition temperature. As more design engineers became more familiar with that term and its significance to a design, the term *smart polymer* is some applications will be eliminated and replace with terms like *working smartly with a polymer.*

We have not addressed the versatility of these smart materials. One such example may involve the smart shock absorbers. Two literature sources discuss the current research that is being focused on vibration suppression in automobiles using smart shock absorbers.[48,49] These smart shock absorbers were developed by Toyota and consisted of multilayer piezoelectric ceramics. These multilayer stacks are positioned near each wheel. After analyzing the vibration signals, a voltage is fed back to the actuator stack that responds by pushing on the hydraulic system to enlarge the motion. Signal processors analyzed the acceleration signals from road bumps and responded with a motion that canceled the vibration.[3,17] Such active piezoelectric systems are used to minimize excess vibrations in helicopter blades and in the twin tails of F-18 fighter jets. One Internet source discussed the work at the University of Rochester with smart shock absorbers using electrorheological fluids. The electrorheological fluids would sense the force of a bump and immediately send an electric signal to precisely alter the amount to dampen the force of the bump thus providing a smoother ride. In an engine mount, electrorheological fluids damp out the vibrations of the engine thus reducing wear and tear on the vehicle. Another application of electrorheological fluids is in the clutches to reduce the wear between the plates as a driver shifts gears. This can reduce the maintenance costs of high-duty trucks.[47] Or one can dampen the vibrations of the road, engine mounts, and other sources on an automobile or heavy-duty truck with magnetorheological fluids. The Lord Corporation has developed a series of trademarked fluids and systems known as Rheonetic that are commercially available for vibration control as well as for noise suppression.[49]

17 FUTURE

The future of smart materials and structures is wide open. The use of smart materials in a product and the type of smart structures that one can design is only limited to one's talents, capabilities, and ability to "think out of the box."

In an early work[5] and as part of the short courses taught there were discussions pertaining to future considerations. A lot of the brainstorming that resulted from these efforts is now being explored. And there are some that were in the conceptual stage are moving forward. Look at the advances made with the automobile and what information and comforts it provides through smart materials and structures.

We can take automobiles to a garage for service and it is hooked up to a diagnostic computer and the mechanic is told what is wrong with the car. Or we have a light on the dashboard that signals "maintenance required." Would it not be better for the light to inform us as to the exact nature of the problem and the severity of it? This approach mimics a cartoon that appeared several years ago of an air mechanic near a plane in a hanger. The plane says "Ouch" and the mechanic says "Where do you hurt?"

One application of smart materials and their application is the earlier work mentioned of using a piezoelectric ink jet printer to serve as a chemical delivery to print organic light-emitting polymers in a fine detail on various medium. Why not take the same application to synthesize smaller molecules? With the right set one could synthesize smaller molecules in significant amounts for characterization and evaluation and in such a way that we could perform design of experiments with relative easy.

A topic of research that was in the literature a few years ago was “smart clothes” or “wearable computers” being studied at MIT. The potential of this concept is enormous. This sounds wonderful as long as we learn how to work smarter not longer.

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CHAPTER 14

OVERVIEW OF CERAMIC MATERIALS, DESIGN, AND APPLICATION

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1 INTRODUCTION

Engineering ceramics possess unique combinations of physical, chemical, electrical, optical, and mechanical properties. Utilizing the gains in basic materials science understanding and advances in processing technology accrued over the past half century, it is now frequently possible to custom tailor the chemistry, phase content, and microstructure to optimize, applications-specific combinations of properties in ceramics (which includes glasses, single crystals, and coatings technologies, in addition to bulk polycrystalline materials). This capability in turn has led to many important, new applications of these materials over the past few decades. Indeed, in many of these applications the new ceramics and glasses are the key enabling technology.

Ceramics include materials that have the highest melting points, highest elastic modulii, highest hardness, highest particulate erosion resistance, highest thermal conductivity, highest optical transparency, lowest thermal expansion, and lowest chemical reactivity known. Counterbalancing these beneficial factors are brittle behavior and vulnerability to thermal shock and impact. Over the past three decades major progress has been made in learning how to design to mitigate the brittleness and other undesirable behaviors associated with ceramics

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Handbook of Materials Selection. Edited by Myer Kutz

and glasses. Consequently, many exciting new applications for these materials have emerged over the past several decades.

Among the major commercial applications for these materials are:

- Passive electronics (capacitors and substrates)
- Optronics/photonics (optical fibers)
- Piezoceramics (transducers)
- Mechanical (bearings, cutting tools)
- Biomaterials (hard tissue replacement)
- Refractories (furnace linings, space vehicle thermal protection)
- Electrochemical (sensors, fuel cells)
- Transparencies (visible, radar)

This chapter will provide a brief overview of how ceramics are processed and the ramifications of processing on properties. Next a short discussion of the special issues that one encounters in mechanical design with brittle materials is provided. Short reviews of several of the above engineering applications of ceramics and glasses, which discuss some of the specific combinations of properties that have led design engineers to the selected material(s), follow. A section on how to obtain information on materials sources and information is provided. Tables listing typical properties of candidate materials for each set of applications are included throughout. Finally, some areas of future potential will be discussed.

2 PROCESSING OF ADVANCED CERAMICS

The production of utilitarian ceramic artifacts via the particulate processing route outlined in Fig. 1 actually commenced about 10,000 years ago.[1] Similarly, glass melting technology goes back about 3500 years, and as early as 2000 years ago optical glass was being produced.[1] While many of the basic unit processes for making glasses and ceramics are still recognizable across the millennia, the level of sophistication in equipment, process control, and raw material control have advanced by "lightyears." In addition, the past 50 years has created a fundamental understanding of the materials science principles that underlie the processing–microstrucure–property relationships. Additionally, new materials have been synthesized that possess extraordinary levels of performance for specific applications. These advances have led to the use of advanced ceramics and glasses in roles that were unimaginable 50 or 60 years ago. For example, early Egyptian glass ca. 2000 BC had an optical loss of $\sim 10^7$ dB/km, compared to an optical loss of $\sim 10^{-1}$ in mid-1980s glass optical fibers,[2] a level of performance that has facilitated the fiber-optic revolution in telecommunications. Similarly, the invention of barium titanate and lead zirconate titanate ceramics, which have much higher piezoelectric moduli and coupling coefficients than do naturally occurring materials, has enabled the existence of modern sonar and medical ultrasound imaging.[3]

The processing of modern ceramics via the particulate route, shown in Fig. 1, is the way that ~99% of all polycrystalline ceramics are manufactured. Other techniques for producing polycrystalline ceramics, such as chemical vapor dep-

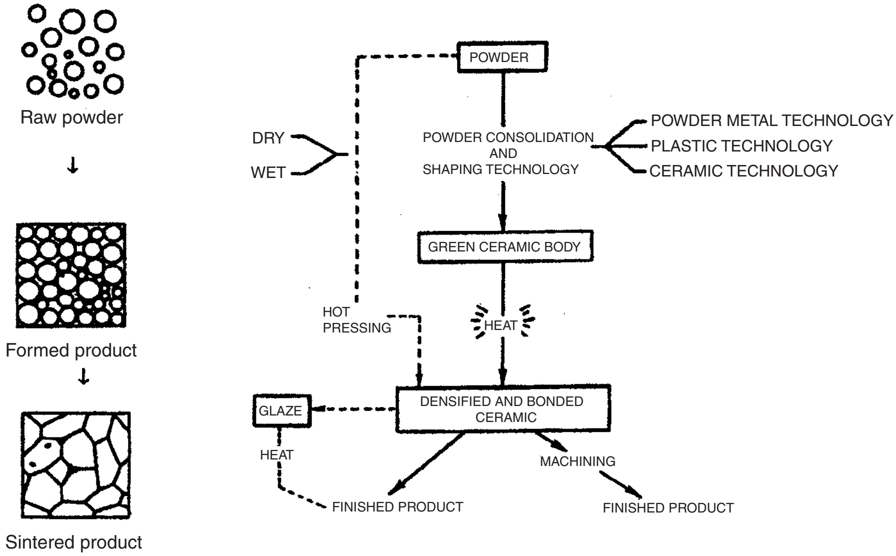

Fig. 1 Processing of polycrystalline ceramics via the particular route.

osition[4] or reaction forming[5] are of growing importance but still represent a very small fraction of the ceramic industry. There are three basic sets of unit processes in the particulate route (and each of these three sets of processes may incorporate dozens of subprocesses). The first set of processes involve powder synthesis and treatment. The second set of processes involve the consolidation of the treated powders into a shaped perform, known as a "green" body. The green body typically contains about 50 vol % porosity and is extremely weak. The last set of unit processes utilize heat, or heat and pressure combined, to bond the individual powder particles, remove the free space and porosity in the compact via diffusion, and create a fully dense, well-bonded ceramic with the desired microstructure.[6] If only heat is used, this process is called sintering. If pressure is also applied, the process is than referred to as hot pressing (unidirectional pressure) or hot isostatic pressing [(HIP), which applies uniform omnidirectional pressure].

Each of the above steps can introduce processing flaws that can diminish the intrinsic properties of the material. For example, chemical impurities introduced during the powder synthesis and treatment steps may adversely effect the optical, magnetic, dielectric, or thermal properties of the material. Alternatively, the impurities may segregate in the grain boundary of the sintered ceramic and negatively effect its melting point, high temperature strength, dielectric properties, or optical properties. In green-body formation, platey or high aspect ratio powders may align with a preferred orientation, leading to anisotropic properties. Similarly, hot pressing may impose anisotropic properties on a material. Since ceramics are not ductile materials, they can (usually) not be thermomechanically modified after primary fabrication. Thus, the specific path by which a ceramic component is fabricated can profoundly effect its properties. The properties encountered in a complex shaped ceramic part are often quite different than those encountered in a simply shaped billet of material. This is an important point of which a design engineer specifying a ceramic component needs to be constantly mindful.

3 BRITTLENESS AND BRITTLE MATERIALS DESIGN

Even when ceramics are selected for other than mechanical applications, in most cases some level of strength and structural integrity are required. It is therefore necessary to briefly discuss the issue of brittleness and how one designs with brittle materials before proceeding to discuss applications and the various ceramic and glass materials families and their properties.

The main issues in designing with a brittle material are that a very large scatter in strength (under tensile stress), a lack of capacity for mitigating stress concentrations via plastic flow, and relatively low energy absorption prior to failure dominate the mechanical behavior. Each of these issues is a result of the presence of one or more flaw distribution within or at the surface of the ceramic material and/or the general lack of plastic flow available in ceramics. As a consequence, ceramic and glass components that are subjected to tensile stresses are not designed using a single valued strength (*deterministic design*) as commonly done with metals. Rather, ceramic components are designed to a specified probability of failure (*probabilistic design*) that is set at acceptably low values. The statistics of failure of brittle materials whose strength is determined by a population of varying sized flaws is similar to modeling the statistics of a chain failing via its weakest link. The statistics utilized are known as Weibull statistics. A Weibull probability of failure distribution is characterized by two parameters, the characteristic stress and the Weibull modulus.[7] Computer programs for incorporating Weibull statistical distributions into finite-element design codes have been developed that facilitate the design of ceramic components optimized for low probabilities of failure.[8] The effectiveness of such probabilistic design methodology has been demonstrated by the reliable performance of ceramics in many highly stressed structural applications, such as bearings, cutting tools, turbocharger rotors, missile guidance domes, and hip prosthesis.

Flaws (strength-limiting features) can be intrinsic or extrinsic to the material and processing route by which a test specimen or a component is made. Intrinsic strength-limiting flaws are generally a consequence of the processing route and may include features such as pores, aggregations of pores, large grains, agglomerates, and shrinkage cracks. While best processing practices will eliminate or reduce the size and frequency of many of these flaws, it is inevitable that some will still persist. Extrinsic flaws can arise from unintended foreign material entering the process stream, i.e., small pieces of debris from the grinding media or damage (cracks) introduced in machining a part to final dimensions. Exposure to a service environment may bring new flaw populations into existence, i.e., oxidation pits on the surface of nonoxide ceramics exposed to high temperatures or may cause existing flaws to grow larger as in the case of static fatigue of glass. In general, one can have several flaw populations present in a component at any time, and the characteristics of each population may change with time. As a consequence of these constantly changing flaw populations, at the present time the state of the art in life prediction of ceramic components for use in extreme environments significantly lags the state of the art in component design.

As in most fields of engineering, there are some rules of thumb that one can apply to ceramic design.[9] While these are not substitutes for a carefully executed probabilistic finite-element design analysis, they are very useful in spotting pitfalls and problems when a full-fledged design cannot be executed due to financial or time constraints.

Rules of Thumb for Design with Brittle Materials

1. Point loads should be avoided to minimize stress where loads are transferred. It is best to use areal loading (spherical surfaces are particularly good); line loading is next best.
2. Structural compliance should be maintained by using compliant layers, springs or radiusing of mating parts (to avoid lockup).
3. Stress concentrators—sharp corners, rapid changes in section size, undercuts and holes—should be avoided or minimized. Generous radiuses and chamfers should be used.
4. The impact of thermal stresses should be minimized by using the smallest section size consistent with other design constraints. The higher the symmetry the better (a cylinder will resist thermal shock better than a prism), and breaking up complex components into subcomponents with higher symmetry may help.
5. Components should be kept as small as possible—the strength and probability of failure at a given stress level are dependent on size; thus minimizing component size increases reliability.
6. The severity of impact should be minimized. Where impact (i.e., particulate erosion) cannot be avoided, low angle impacts (20°–30°) should be designed for. Note this is very different than the case of metals, where minimum erosion is at 90°.
7. Avoid surface and subsurface damage. Grinding should be done so that any residual grinding marks are parallel, not perpendicular, to the direction of principal tensile stress during use. Machining induced flaws are often identified to be the strength-limiting defect.

4 APPLICATIONS

The combinations of properties available in many advanced ceramics and glasses provide the designers of mechanical, electronic, optical, and magnetic systems a variety of options for significantly increasing systems performance. Indeed, in some cases the increase in systems performance is so great that the use of ceramic materials is considered an enabling technology. In the applications examples provided below the key properties and combinations of properties required will be discussed, as well as the resultant systems benefits.

4.1 Ceramics in Wear Applications

In the largest number of applications where modern ceramics are used in highly stressed mechanical applications, they perform a wear resistance function. This is true of silicon nitride used as balls in rolling element bearings, silicon carbide journal bearings or water pump seals, alumina washers in faucets and beverage dispensing equipment, silicon nitride and alumina-based metal cutting tools, zirconia fuel injector components, or boron carbide sand blast nozzles, to cite some typical applications and materials.

Wear is a systems property rather than a simple materials property. As a systems property, wear depends upon what material is rubbing, sliding, or rolling over what material, upon whether the system is lubricated or not, upon what the lubricant is, and so forth. To the extent that the wear performance of a material can be predicted, the wear resistance is usually found to be a complex function of several parameters. Wear of ceramic materials is often modeled using an abrasive wear model where the material removed per length of contact with the abrasive is calculated. A wide variety of such models exist, most of which are of the form:

$$V \propto P^{0.8} {K_{\mathrm{Ic}}}^{-0.75} H^{-0.5} N \tag{1}$$

where V is the volume of material worn away, P is the applied load, K_{Ic} is the fracture toughness, H is the indentation hardness, and N is the number of abrasive particles contacting the wear surface per unit length. Even if there are no external abrasives particles present, the wear debris of the ceramics themselves act as abrasive particles. Therefore, the functional relationships that predict that wear resistance should increase as fracture toughness and hardness increase are, in fact, frequently observed in practice.

Even though the point contacts that occur in abrasive wear produce primarily hertzian compressive stresses, in regions away from the hertzian stress field tensile stresses will be present and strength is, thus, a secondary design property. In cases where inertial loading or weight is a design consideration, density may also be a design consideration. Accordingly, Table 1, lists the fracture toughness, hardness, Young's modulus, four-point modulus of rupture (MOR) in tension, and the density for a variety of advanced ceramic wear materials. Several successful applications of ceramics to challenging wear applications are described below.

Bearings

Rolling element bearings, for use at very high speeds or in extreme environments, are limited in performance by the density, compressive strength, corrosion resistance, and wear resistance of traditional high-performance bearing steels. The key screening test to assess a material's potential as a bearing element is

Table 1 Key Properties for Wear-Resistant Ceramics

Material	K_{Ic}(MPa · m$^{1/2}$)	H (kg/mm^2)	E (GPa)	MOR (MPa)	ρ (g/cm^3)
Al_2O_3 99%	3.9–4.5	1900	360–395	350–560	3.9
B_4C	—	3000	445	300–480	2.5
Diamond	6–10	8000	800–925	800–1400	3.5
SiC	2.6–4.6	2800	380–445	390–550	3.2
Si_3N_4	4.2–7	1600	260–320	450–1200	3.3
TiB_2	5–6.5	2600	550	240–400	4.6
ZrO_2 (Y-TZP)	7–12	1000	200–210	800–1400	5.9

rolling contact fatigue (RCF). RCF tests on a variety of alumina, SiC, Si_3N_4, and zirconia materials, at loads representative of high-performance bearings, demonstrated that only fully dense silicon nitride (Si_3N_4) could out-perform bearing steels.[10] This behavior has been linked to the high fracture toughness of silicon nitride, which results from a unique ‘‘self-reinforced’’ microstructure, combined with a high hardness. Additionally, the low density of silicon nitride creates a reduced centrifugal stress on the outer races at high speeds. Fully dense Si_3N_4 bearing materials have demonstrated RCF lives 10 times that of high-performance bearing steel. This improved RCF behavior translates into DN (DN = bearing bore diameter in millimeters × shaft rpm) ratings for hybrid ceramic bearings (Si_3N_4 balls running in steel races, the most common ceramic bearing configuration), about 50% higher than the DN rating of steel bearings. Other benefits of silicon nitride hybrid bearings include an order of magnitude less wear of the inner race, excellent performance under marginal lubrication, survival under lubrication starvation conditions, lower heat generation than comparable steel bearings, and reduced noise and vibration.

Another important plus for Si_3N_4 is its failure mechanism. When Si_3N_4 rolling elements fail, they do not fail catastrophically; instead they spall—just like bearing steel elements (though by a different microstructural mechanism). Thus, the design community only had to adapt their existing practices, instead of developing entirely new practices to accommodate new failure modes. The main commercial applications of silicon nitride bearing elements are listed in Table 2.

Cutting Tool Inserts

While ceramic cutting tools have been in use for over 60 years, it is only within the past two decades that they have found major application, principally in turning and milling cast-iron and nickel-based superalloys and finishing of hardened steels. In these areas ceramics based on aluminum oxide and silicon nitride significantly outperform cemented carbides and coated carbides. High-speed cutting tool tips can encounter temperatures of 1000°C or higher. Thus, a key property for an efficient cutting tool is hot hardness. Both the alumina and Si_3N_4 families of materials retain a higher hardness at temperatures between 600 and 1000°C than either tool steels or cobalt-bonded WC cermets. The ceramics are also more chemically inert. The combination of hot hardness and chemical inertness means that the ceramics can run hotter and longer with less wear than

Table 2 Commercial Applications of Si_3N_4 Hybrid Bearings

Machine tool spindles	The first and largest application, its main benefits are higher speed and stiffness, hence greater throughput and tighter tolerances
Turbomolecular pump shaft	Presently the industry standard, the main benefits are improved pump reliability and marginal lubrication capability, which provides increased flexibility in pump mounting orientation
Dental drill shaft	The main benefit is sterilization by autoclaving
Aircraft wing flap actuators	Wear and corrosion resistance are the main benefits
In-line skates/mountain bikes	Wear and corrosion resistance are the main benefits
Space Shuttle main engine oxygen fuel pump	Here, the bearing is lubricated by liquid oxygen. Steel bearings are rated for one flight; Si_3N_4 hybrid bearings are rated for five.

the competing materials. Historic concerns with ceramic cutting tools have focused on low toughness, susceptibility to thermal shock, and unpredictable failure times. Improvements in processing, together with microstructural modifications to increase fracture toughness have greatly increased the reliability of the ceramics in recent years

Alumina-based inserts are reinforced (toughened) with zirconia, TiC, or TiN particles or SiC whiskers. The thermal shock resistance of alumina–SiC_w is sufficiently high, so that cooling fluids can be used when cutting Ni-based alloys. Silicon-nitride-based inserts include fully dense Si_3N_4 and SiAlON's, which are solid solutions of alumina in Si_3N_4. Fully dense Si_3N_4 can have a fracture toughness of 6–7 $MPa \cdot m^{1/2}$, almost as high as cemented carbides ($\sim$9 $MPa \cdot m^{1/2}$), a high strength (greater than 1000 MPa) and a low thermal expansion that yields excellent thermal shock behavior. Silicon nitride is the most efficient insert for the turning of gray cast iron and is also used for milling and other interrupted cut operations on gray iron. Because of its thermal shock resistance, coolant may be used with silicon nitride for turning applications. SiAlON's are typically more chemically stable than the Si_3N_4's but not quite as tough or thermal shock resistant. They are mainly used in rough turning of Ni-based superalloys.

Ceramic inserts are generally more costly than carbides (1.5–2 times more), but their metal removal rates are $\sim$3–4 times greater. However, that is not the entire story. Ceramic inserts also demonstrate reduced wear rates. The combination of lower wear and faster metal removal means many more parts can be produced before tools have to be indexed or replaced. In some cases this enhanced productivity is truly astonishing. In the interrupted single-point turning of the outer diameter counterweights on a gray cast-iron crankshaft a SiAlON tool was substituted for a coated carbide tool. This change resulted in the metal removal rate increasing 150% and the tool life increasing by a factor of 10. Each tool now produced 10 times as many parts and in much less time. A gas turbine manufacturer performing a machining operation on a Ni-based alloy using a SiAlON tool for roughing and a tungsten carbide tool for finishing required a total of 5 h. Changing to SiC-whisker-reinforced alumina inserts for both operations reduced the total machining time to only 20 min. This yielded a direct savings of $250,000 per year, freed up 3000 h of machine time per year, and avoided the need to purchase a second machine tool.

Ceramic Wear Components in Automotive and Light-Truck Engines

Several engineering ceramics have combinations of properties that make them attractive materials for a variety of specialized wear applications in automotive engines.

The use of structural ceramics as wear components in commercial engines began in Japan in the early 1980s. Table 3, lists many of the components that have been manufactured, the engine company that first introduced the component, the material, and the year of introduction. In some of these applications several companies have introduced a version of the component into one or more of their engines.

Many of these applications are driven by the need to control the emissions of heavy-duty diesels. Meeting current emissions requirements creates conditions within the engine fuel delivery system that increase wear of lubricated steel

Table 3 Ceramic Wear Components in Automotive and Light-Truck Engines

Component	Engine Manufacturer	Engine Type	Ceramic	Year of Introduction
Rocker arm insert	Mitsubishi	SI	Si_3N_4	1984
Tappet	Nissan	Diesel	Si_3N_4	1993
Fuel injector link	Cummins	Diesel	Si_3N_4	1989
Injector shim	Yanmar	Diesel	Si_3N_4	1991
Cam roller	Detroit Diesel	Diesel	Si_3N_4	1992
Fuel injector timing plunger	Cummins	Diesel	ZrO_2	1995
Fuel pump roller	Cummins	Diesel	Si_3N_4	1996

SI = spark-ignited engine.

against steel. One of these conditions is increased injection pressure, another is an increase in the soot content of engine lubricating oils. Strategic utilization of ceramic components within the fuel delivery systems of many heavy-duty truck engines has enabled the engines to maintain required performance for warranties of 500,000 miles and more. The fuel injector link introduced by Cummins in 1989 is still in production. Well over a million of these components have been manufactured. And many of these have accumulated more than a million miles of service with so little wear that they can be reused in engine rebuilds. In a newer model electronic fuel injector, Cummins introduced a zirconia timing plunger. The part has proved so successful that a second zirconia component was added to the timing plunger assembly several years later. Increasingly stringent emissions requirements for heavy diesels has increased the market for ceramic components in fuel injectors and valve train components. Many of these heavy-duty engine parts are manufactured at rates of 20,000 up to 40,000 per month.

Perhaps the largest remaining problem for this set of applications is cost. Ceramic parts are still more expensive than generally acceptable for the automotive industry. Reluctance of designers to try ceramic solutions still exists, but it is greatly diminishing thanks to the growing list of reliable and successful application of structural ceramic engine components.

4.2 Thermostructural Applications

Due to the nature of their chemical bond, many ceramics maintain their strength and hardness to higher temperatures than metals. For example, at temperatures above 1200°C, silicon carbide and silicon nitride ceramics are considerably stronger than any superalloy. As a consequence, structural ceramics have been considered and utilized in a number of demanding applications where both mechanically imposed tensile stresses and thermally imposed tensile stresses are present. One dramatic example is the ceramic (silicon nitride) turbocharger that has been in commercial production for automobiles in Japan since 1985. Over one million of these have been manufactured and driven with no recorded failure. This is a very demanding application, as the service temperature can reach 900°C, stresses at ~700°C can reach 325 MPa, and the rotor must also endure oxidative and corrosive exhaust gases that may contain erosion inducing rust and soot particles. Silicon nitride gas turbine nozzle vanes have been flying for several years in aircraft auxiliary power units. Other applications include heat exchangers, and hot gas valving. Recently, ceramic matrix composites have been introduced as disks for disk breaks in production sports cars by two European manufacturers. A major future market for structural ceramics may be high-performance automotive valves. Such valves are currently undergoing extensive, multiyear fleet tests in Germany.

This class of applications requires a focus on the strength, Weibull modulus, m (the higher the m, the narrower the distribution of observed strength values), thermal shock resistance, and often the stress rupture (strength decrease over time at temperature) and/or creep (deformation with time at temperature) behavior of the materials. Indeed, as shown in Fig. 2, the stress rupture performance of current structural ceramics represents a significant jump in materials performance over superalloys.

The thermal shock resistance of a ceramic is a systems property rather than a fundamental materials property. Thermal shock resistance is given by the maximum temperature change a component can sustain, ΔT.

$$\Delta T = \frac{\sigma(1 - \mu)}{\alpha E} \frac{k}{r_m h} S \tag{2}$$

where σ is strength, μ is Poisson's ratio, α is the coefficient of thermal expansion (CTE), E is Young's modulus, k is thermal conductivity, r_m is the half-thickness for heat flow, h is the heat transfer coefficient, and S is a shape factor totally dependent on component geometry.[11] Thus it can be seen that thermal shock resistance, ΔT, is made up of terms wholly dependent on materials properties, dependent on heat transfer conditions, and on geometry. It is the role of the ceramic engineer to maximize the former and of the design engineer to maximize the later two terms. It has become usual practice to report the materials-related thermal shock resistance as the instantaneous thermal shock parameter, R, which is equal to

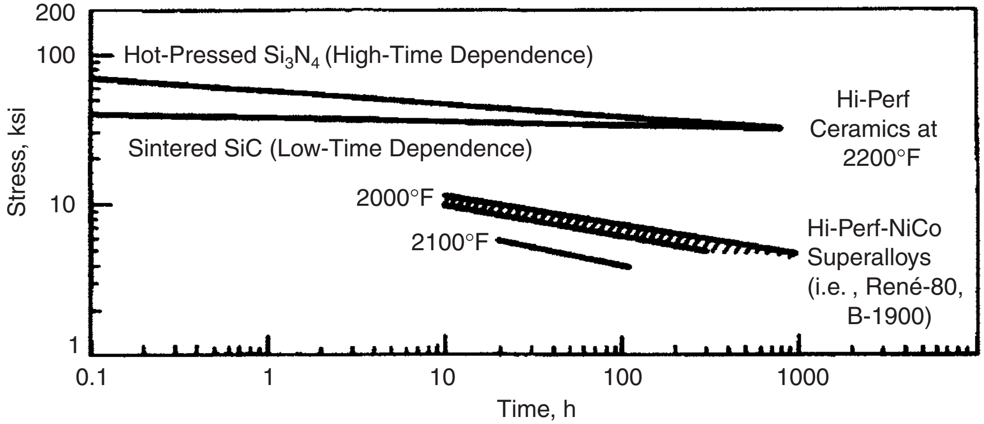

Fig. 2 Stress rupture performance of nonoxide structural ceramics compared to superalloys (oxidizing atmosphere).

Table 4 Calculated Thermal Shock Resistance of Various Ceramics

Material	σ (MPa)	μ	CTE (cm/cm K)	E (GPa)	R (K)
Al_2O_3 (99%)	345	0.22	7.4×10^{-6}	375	97
AlN	350	0.24	4.4×10^{-6}	350	173
SiC (sintered)	490	0.16	4.2×10^{-6}	390	251
PSZ	1000	0.3	10.5×10^{-6}	205	325
Si_3N_4 (sintered)	830	0.3	2.7×10^{-6}	290	742
LAS (glass CERAMIC)	96	0.27	0.5×10^{-6}	68	2061
Al-titanate	41	0.24	1.0×10^{-6}	11	2819

$$R = \frac{\sigma(1 - \mu)}{\alpha E} \tag{3}$$

The value of R for selected ceramics is presented in Table 4. Another frequently used parameter is R', the thermal shock resistance where some heat flow occurs: R' is simply R multiplied by the thermal conductivity, k. For cases where heat transfer environments are complex, Ref. 12 lists 22 figures of merit for selecting ceramics to resist thermal stress.

4.3 Corrosion Resistance

Many advanced structural ceramics such as alumina, silicon nitride, or SiC have strong atomic bonding that yields materials that are highly resistant to corrosion by acidic or basic solutions at room temperature (the notable exception being glass or glass-bonded ceramics attacked by HF). This corrosion resistance has led to many applications. Carbonated soft drinks are acidic, and alumina valves are used to meter and dispense these beverages at refreshment stands. The chemical industry utilizes a wide variety of ceramic components in pumps and valves for handling corrosive materials. For example, the outstanding corrosion resistance of fully dense SiC immersed in a variety of hostile environments is given in Table 5. There are many cases where corrosion and particulate wear are superimposed, as in the handling of pulp in papermaking or transporting slurries in mineral processing operations, and ceramics find frequent application in such uses.

Table 5 Weight Loss of Fully Dense SiC in Acids and Bases

Reagent (wt %)	Test Temperature (°C)	Weight Loss (mg/cm^2 yr)
98% H_2SO_4	100	1.5
50% NaOH	100	2.5
53% HF	100	<0.2
85% H_3PO_4	100	<0.2
45% KOH	100	<0.2
25% HCl	100	<0.2
10% HF + 57% HNO_3	25	<0.2

Specimens submerged 125 to 300 h, continuously stirred.
Data Courtesy of ESK-Wacker, Adrian, MI.

4.4 Passive Electronics

The role of passive electronics is to provide insulation (prevent the flow of electrons) either on a continuous basis (as in the case of substrates or packages for microelectronics) or on an intermittent basis as is the case for ceramic capacitors (which store electric charge and hence need a high polarizability). These two applications constitute two of the largest current markets for advanced ceramics. For electronic substrates and packages key issues include the minimization of thermal mismatch stresses between the Si (or GaAS) chip and the package material (so the CTE will be important) and dissipation of the heat generated as electrons flow through the millions of transistors and resistors that comprise modern microelectronic chips; hence the thermal conductivity is a key property. All other things being equal, the delay time for electrons to flow in the circuit is proportional to the square root of the dielectric constant of the substrate (or package) material. Additionally, the chip or package must maintain its insulating function, so resistivities of $>10^{14}$ are required. Most high-performance packages for computer chips are alumina. With the advent of microwave integrated circuits (e.g., cell phones) aluminum nitride substrates are beginning to be utilized for high thermal conductivity. The environmental drawbacks to machining BeO has tended to favor the use of AlN to replace or avoid the use of BeO. Synthetic diamond is an emerging substrate material for special applications. Isotopically "pure" synthetic, single-crystal diamond has values of thermal conductivity approaching 10,000 W/mK. Typical values of the above properties for each of these materials is given in Table 6, along with selected properties of silicon for comparison. For design purposes exact values for specific formulations of the materials should be obtained from the manufacturers.

Over a billion ceramic capacitors or multilayer ceramic capacitors (MLCC) are made every day.[13] Since electrons do not flow through capacitors, they are considered passive electronic components. However, the insulators from which ceramic capacitors are made polarize thereby separating electric charge. This separated charge can be released and flow as electrons, but the electrons do not flow through the dielectric material of which the capacitor is composed. Thus, the materials parameter, which determines the amount of charge that can be stored, the dielectric constant, *k*, is the key parameter for design and application. Table 7 lists the approximate dielectric constant at room temperature for several families of ceramics used in capacitor technology. The dielectric constant varies

Table 6 Key Properties for Electronic Substrates and Packages

Material	CTE (10^{-6}/K)	Therm. Cond. (W/mK)	Resistivity	Dielectric Const.
Al_2O_3 (96%)	6.8	26	$>10^{14}$	9.5
Al_2O_3 (99%)	6.7	35	$>10^{14}$	10
AlN	4.5	140–240	$>10^{14}$	9
BeO	6.4	250	$>10^{14}$	6.5
Diamond	2	2000	$>10^{14}$	5.5
Silicon	2.8	150		

Note: CTE and thermal conductivity are at room temperature, and the dielectric constant is at 1 MHz.

Table 7 Dielectric Constants for Various Ceramic Capacitor Materials

Material	Dielectric Constant at RT
Tantalum oxide (Ta_2O_5)	~25
Barium titinate	~5,000
Barium–zirconium titinate	~20,000
Lead–zirconium titinate (PZT)	~2,000
PZT with W or Mg additives	~9,000
Lead magnesiun niobate (PMN)	~20,000
Lead zinc niobate (PZN)	~20,000

with both temperature and frequency. Thus, for actual design precise curves of materials performance over a relevant range of temperatures and frequencies are often utilized. Many ceramics utilized as capacitors are ferroelectrics, and the dielectric constant of these materials is usually a maximum at or near the Curie temperature.

4.5 Piezoceramics

Piezoceramics are a multi-billion dollar market.[14] Piezoceramics are an enabling material for sonar systems, medical ultrasonic imaging, micromotors and micropositioning devices, the timing crystals in our electronic watches, and numerous other applications. A piezoelectric material will produce a charge (or a current) if subjected to pressure (the *direct* piezoelectric effect) or, if a voltage is applied, the material will produce a strain (the *converse* piezoelectric effect). Upon the application of a stress, a polarization charge, P, per unit area is created that equals $d\sigma$, where σ is the applied stress and d is the piezoelectric modulus. This modulus, which determines piezoelectric behavior, is a third-rank tensor[15] that is thus highly dependant on directions along which the crystal is stressed. For example, a quartz crystal stressed in the [100] direction will produce a voltage, but one stressed in the [001] direction will not. In a polycrystalline ceramic the random orientation of the grains in an as-fired piezoceramic will tend to minimize or zero out any net piezoelectric effects. Thus, polycrystalline piezoceramics have to undergo a postsintering process to align the electrically charged dipoles within the polycrystalline component. This process is known as poling and it requires the application of a very high electric field. If the piezoceramic is taken above a temperature, known as the Curie temperature, a phase transformation occurs and piezoelectricity will disappear. The piezoelectric modulus and the Curie temperature are thus two key materials selection parameters for piezoceramics.

The ability of piezoceramics to almost instantaneously convert electrical current to mechanical displacement, and vice versa, makes them highly usefull as transducers. The efficiency of conversion between mechanical and electrical energy (or the converse) is measured by a parameter known as the coupling coefficient. This is a third key parameter that guides the selection of piezoelectric materials.

Although piezoelectricity was discovered by Pierre and Jacques Curie in 1880, piezoceramics were not widely utilized until the development of polycrystalline barium titanate in the 1940s and lead zirconate titanates (PZT) in the 1950s. Both of these materials have high values of *d* and thus develop a high voltage for a given applied stress. PZT has become widely used because in addition to a high *d* value, it also has a very high coupling coefficient. Sonar, in which ultrasonic pulses are emitted and reflected ‘‘echoes’’ are received, is used to locate ships, fish, and map the ocean floor by navies, fishermen, and scientists all over the globe. Medical ultrasound utilizes phased arrays of piezoceramic transducers to image organs and fetuses noninvasively and without exposure to radiation. A relatively new application that has found significant use in the microelectronics industry is the use of piezoceramics to drive micropositioning devices and micromotors. Some of these devices can control positioning to a nanometer or less. Piezoceramic transducers combined with sophisticated signal detection and generation electronics to create ‘‘active’’ noise and vibration damping devices. In such devices the electronics detect and quantify a noise spectrum and then drive the transducers to provide a spectrum 180° out of phase with the noise, thereby, effectively canceling it.

Many of the current high-performance applications of piezoceramics are based on proprietary modifications of PZT, which contain additions of various dopants or are solid solutions with perovskite compounds of Pb with Mg, Mn, Nb, Sn, Mo, or Ni. Table 8, lists the range of several key piezoceramic selection parameters for proprietary PZT compositions from one manufacturer.

4.6 Transparencies

Transparent ceramics (which include glasses, single-crystal, and polycrystalline ceramics) have been used as optical transparencies or lenses for millennia. Glass windows were in commercial production in first century Rome, but it was not until the 1800s, with the need for precision optics for microscopes, telescopes, and ophthalmic lenses, that glasses and other optical materials became the object of serious scientific study. As noted in the introduction, progress in glass science and technology, coupled with lasers, has led to the current broadband digital data transmission revolution via optical fibers. Various ceramic crystals are used as laser hosts and specialty optical lenses and windows. A significant fraction of supermarket scanner windows combine the scratch resistance of sapphire (single-crystal alumina) with its ability to transmit the red laser light that we see at the checkout counter. While such windows are significantly more costly than glass, their replacement rate is so low that they have increased profitability for several supermarket chains. For the same reason the crystal in many high-end watches are scratch-resistant manmade sapphire. Polycrystalline translucent (as opposed to fully transparent) alumina is used as containers (envelops) for the sodium vapor lamps that light our highways and industrial sites.

Table 8 Key Properties for PZT Based Piezoceramics

Material	Piezoelectric Modulus, d_{33}	Curie Temp (°C)	Coupling Coefficent, k_{33}
A	226×10^{-12} m/V	320	0.67
B	635×10^{-12} m/V	145	0.68
C	417×10^{-12} m/V	330	0.73

Not all windows have to pass visible light. Radar or mid- to far-infrared transparencies look opaque to the human eye but are perfectly functional windows at their design wavelengths. The most demanding applications for such transparencies is for the guidance domes of missiles. Materials that can be used for missile radomes include slip-cast fused silica, various grades of pyroceram (glass ceramics), and silicon-nitride based materials. Infrared (IR) windows and missle domes include MgF_2 and ZnSe. Requirements exist for having missile guidance domes that can transmit in the visible, IR, and radar frequencies (multimode domes). Ceramic materials that can provide such functionality include sapphire and aluminum oxynitride spinel (AlON). In addition to optical properties missile domes must be able to take high aerothermal loading (have sufficient strength) and be thermal shock resistant (a high-speed missile encountering a rain cloud can have an instant ΔT of minus several hundred degrees kelvin).

Key properties for visible and IR optical materials include the index of refraction, n (which will be a function of wavelength), and absorption or loss. For radar transparencies key parameters are dielectric constant (which can be thought of as analogous to the index of refraction) and dielectric loss.

5 INFORMATION SOURCES

5.1 Manufacturers and Suppliers

There are hundreds of manufacturers of advanced ceramics and glasses. Locating ones that either already have the material that is needed, and can produce it in the configuration required, can be a daunting task. There are two resources published annually that make this task much easier. The American Ceramic Society publishes a directory of suppliers of materials, supplies, and services that can help locate such information quickly. It is called *Ceramic Source.* This directory can also be accessed on the worldwide web at www.ceramics.org. A similar *Buyers Guide* is published by *Ceramic Industry Magazine,* and this can also be viewed online at www.ceramicindustry.com. Once a likely source for your need has been identified, a visit to the supplier's web site can often provide a great deal of background information and specific data, which can make further contacts with the supplier much more meaningful and informative.

5.2 Data

Manufacturer's literature both hardcopy and posted on the worldwide web, are invaluable sources of data. The handbooks, textbooks, and encyclopedias listed below are also excellent sources of data. However, before committing to a finalized design or to production, it is advisable to develop one's own test data in conformance with you own organization's design practice. Such data should be acquired from actual components made by the material, processing route, and manufacturer that have been selected for the production item.

ASM, *Ceramics and Glasses, Vol 4, Engineered Materials Handbook,* ASM International, Materials Park, OH, 1991.

J. F. Shackelsford, W. Alexander, and J. Park (eds.), *Materials Science and Engineering Handbook,* 2nd ed., CRC Press, Boca Raton, FL, 1994.

C. X. Campbell and S. K. El-Rahaiby (eds.), *Databook on Mechanical and Thermophysical Properties of Whisker-Reinforced Ceramic Matrix Composites,* Ceramics Information Analysis Center, Purdue University, W. Lafayette, IN, and The American Ceramic Society, Westerville, OH, 1995.

R. J. Brook (ed.), *Concise Encyclopedia of Advanced Ceramic Materials,* Pergamon Press, Oxford, 1991.

5.3 Standards and Test Methods

To reliably design, procure materials, and assure reliability, it is necessary to have common, agreed-upon, and authoritative test standards, methods, and practices. Institutions such as the American Society for Testing of Materials (ASTM), the Japanese Institute for Standards (JIS), the German Standards Organization (DIN), and the International Standards Organization (ISO) all provide standards for there various constituencies. The following are a sampling of standards available from the ASTM and JIS for advanced ceramics and ceramic matrix composites. One can reach these organizations at the following addresses.

American Society for Testing Materials (ASTM), 100 Barr Harbor Drive, Conshohocken, PA 19428-2959:

- C-177-85(1993), Test Method for Steady State Heat Flux and Thermal Transmissionby Means of the Gradient-Hot-Plate Apparatus
- C-1161-90, Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature
- C-1211-92, Test Method for Flexural Strength of Advanced Ceramics at Elevated Temperature
- C-1259-94, Test Method for Dynamic Young's Modulus, Shear Modulus and Poisons Ratio for Advanced Ceramics by Impulse Excitation of Vibration
- C-1286-94, Classification for Advanced Ceramics
- C-1292-95A, Test Method for Shear Strength of Continuous Fiber-Reinforced Ceranic Composites (CFCCs) at Ambient Temperatures
- C-1337-96, Test Method for Creep and Creep-Rupture of CFFFs under Tensile Loading at Elevated Temperature
- C-1421-99, Standard Test Method for Determination of Fracture Toughness of Advanced Ceramics at Ambient Temperature
- C-1425-99, Test Method for Interlaminar Shear Strength of 1-D and 2D CFCCs at Elevated Temperature
- E-228-85(1989), Test Method for Linear Thermal Expansion of Solid Materials with aVitreous Silica Dilatometer
- E-1269-94, Test Method for Determining Specific Heat Capacity by DifferentialScanning Calorimetry
- E-1461-92, Test Method for Thermal Diffusion of Solids by the Flash Method

The Japanese Standards Association, 1-24, Akasaka 4, Minato-ku, Tokyo 107 Japan:

- Testing Methods for Elastic Modulus of High Performance Ceramics at ElevatedTemperatures; JIS R 1605-(1989)
- Testing Methods for Tensile Strength of High Performance Ceramics at Room and Elevated Temperatures; JIS R 1606-(1990)
- Testing Methods for Fracture Toughness of High Performance Ceramics; JIS R 1607-(1990)
- Testing Methods for Compressive Strength of High Performance Ceramics; JIS R 1608-(1990)
- Testing Methods for Oxidation Resistance of Non-Oxide of High Performance Ceramics; JIS R 1609-(1990)
- Testing Methods for Vickers Hardness of High Performance Ceramics; JIS R 1610-(1991)
- Testing Methods of Thermal Diffusivity, Specific Heat Capacity, and ThermalConductivity for High Performance Ceramics by Laser Flash Method; JIS R 1611-(1991)

In addition to testing standards, it is possible to obtain standard materials with certified properties to calibrate several of these new standards against your own tests. Such standard materials can be obtained from the National Institute of Science and Technology (NIST). For example, a standard material to calibrate ASTM C-1421-99 has just been made available. Materials standards are not available for all of the above tests.

5.4 Design Handbooks

It has been widely recognized that procedural handbooks that provide methodology on how to design with advanced ceramics and which can provide high quality evaluated design data are sorely needed for ceramic materials. The ceramics matrix composites (CMCs) community has taken the initiative to begin the process of creating such a handbook for its constituency. The activity is sponsored by various U.S. governmental agencies including the Department of Defense, the Department of Energy, The Federal Aviation Administration, and the National Aeronautics and Space Administration and is entitled MIL-Handbook-17. This activity brings together materials suppliers, materials testers, designers, and end users who are engaged in developing a handbook that will provide design tools and guidance, provide guidelines on data generation, documentation, and use, and provide an authoritative source of design quality data. This is a work in progress and its completion is many years off, if ever. Nevertheless, much guidance in design and testing of advanced CMCs has already resulted from this activity. Progress can be followed by periodically accessing the handbook websites at http://mil-17.udel.edu or http://www.materials-sciences.com/MIL17/. Unfortunately, no similar activity exists for monolithic ceramics.

6 FUTURE TRENDS

It has been estimated that in the United States advanced ceramics of the type discussed above are an over \$8-billion-dollar a year industry with a growth rate of ~8% per year.[16]

The largest segment of this growth will come from the electronics area. Not only will there be significant growth in the "traditional" roles of ceramics as insulators, packages, substrates, and capacitors, but structural ceramics will play a major role in the equipment used in semiconductor manufacturing. This trend will be especially driven by the resistance of ceramics such as SiC, AlN, silicon nitride, and alumina to the erosive and corrosive environments within high-energy plasma chambers used in single-wafer processing operations.

The intertwined global issues of energy sufficiency and environmental protection will see commercial use of advanced ceramics in energy systems as diverse as solid oxide fuel cells and pebble-bed modular reactors (nuclear). As more and more industries move toward "green" (pollution free) manufacturing, there will be growth in wear- and corrosion-resistant ceramics for industrial machinery. There will also be substantial growth potential for ceramic filters and membranes. One major environmentally driven opportunity will be particulate traps for diesel trucks and industrial power sources. This technology is just beginning to be commercialized, and it is certain to see rapid growth as emissions requirements for diesel engines grow more stringent. Not all progress in these areas will create increased markets for ceramics; some will reduce them. For example, the rapid growth of energy-efficient light-emitting diode technology for illumination will create a significant growth opportunity for producers of single-crystal SiC substrates and GaN materials. However, this will come at a cost to the ceramics industry of a significant decrease in glass envelopes for incandescent bulbs and fluorescent tubes. Another area of growth will be filters and membranes for filtration of hot or corrosive, or both, gases and liquids.

The explosive growth of fiber-optic- and microwave-based digital communications technology has produced significant opportunities and markets for advanced ceramics and glasses and will continue to do so into the foreseeable future.

Medical applications are sure to grow, both in the area of diagnostics and prosthetics.

At the entrance to the Pohang Steel complex in Pohang, Republic of Korea, is a wonderful sign. It proclaims; "Resources are Limited—Creativity is Unlimited." This thought certainly applies to the global future of advanced ceramics. Creatively utilized advanced ceramics will effectively expand our resources, protect our environment, and create new technological opportunities. The potential opportunities go far beyond the few discussed in this chapter.

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PART 3

FINDING AND MANAGING MATERIALS INFORMATION AND DATA

Handbook of Materials Selection. Edited by Myer Kutz

CHAPTER 15

HOW TO FIND MATERIALS PROPERTIES DATA

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1 INTRODUCTION

Locating information, especially information regarding the physical properties of materials, is often like doing a logic puzzle. First you have an explanation of what you are going to do with limited information. Then you are given a set of clues or data that are known. Starting with what you know, you sort information logically to get to the "correct answer." When you first start solving the logic puzzle, it always seems there are too few clues. This tempts you to make assumptions such as "the character named Chris is female." Making assumptions early in the process often means you end up with the wrong answer. When starting to look for data and other information about materials, it is easy to assume that the information must be in a specific tool or database. It is often easier to assume the data simply does not exist, that it must exist in a specified format to be useful, or that the information must be retrieved from the engineering literature. The two most insidious assumptions are that the information will be published in English and that to contain good data the information must have been published in the last 5 years (OK, maybe 10 years). Falling prey to these assumptions will mean much time is wasted both in the information search process and when developing novel materials.

To solve a logic puzzle, the puzzle solver will take multiple steps and often repeat those steps until the puzzle has been completed (see Fig. 1). The basic process is cyclic with a solution being reached when enough information has been gathered. The solution should always be double-checked before going to the answer key.

Luckily, when you are locating information to solve materials engineering problems you do not rely on just what you have in front of you. In fact, looking for unique data to help you solve your problem is the best way to deal with

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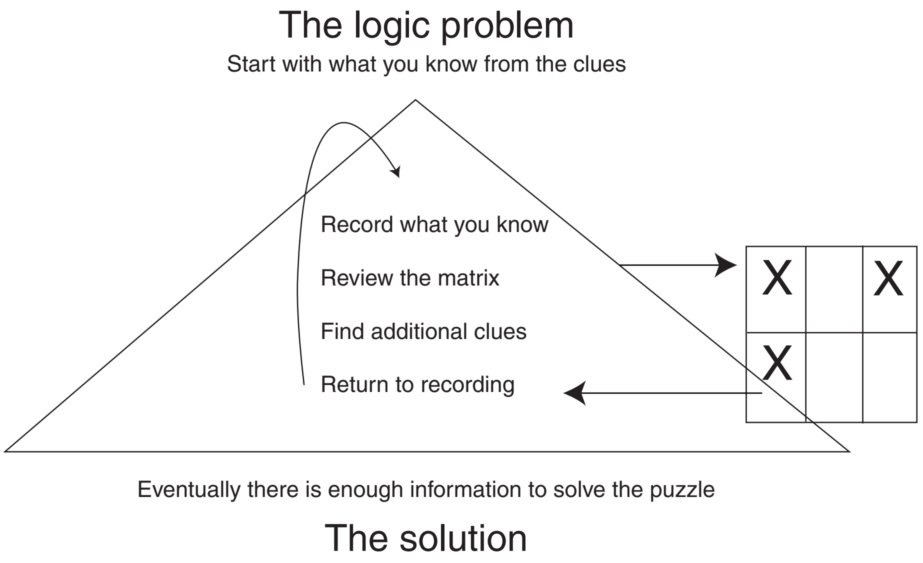


Fig. 1 Solving a logic puzzle.

real-life logic puzzles, especially in materials science. Unfortunately, because you do not have to limit your quest for information to the description provided, the process is a bit more complicated though is similar to solving the logic puzzle. Table 1 shows a "logic" grid that information seekers use subconsciously. This grid shows three logic steps:

Know what your compound/material is and how it will be referred to in the literature.
Know what information is in each data source.
Learn how to use the indexes available in the data sources.

Looking for information is cyclic in nature, with steps being redone until the information needed is gathered (or until you are sure there is no information to find). The solution should be double-checked when possible by using multiple sources. The steps held in common when searching for any information are:

Define the question.
Set up a strategy to locate the needed information.
Use the resources you know best.
Go to less known resources when necessary.
Evaluate the quality of the data/information resource.
Start again, if needed, using the new information to help better define the question.
Know when the information gathered is enough (or probably all there is).

This chapter will discuss these steps in depth making recommendations that will help the materials engineer find data and information more easily and within

Table 1 Resource Grid: Focus on Tools That May Have Information Needed—An Example

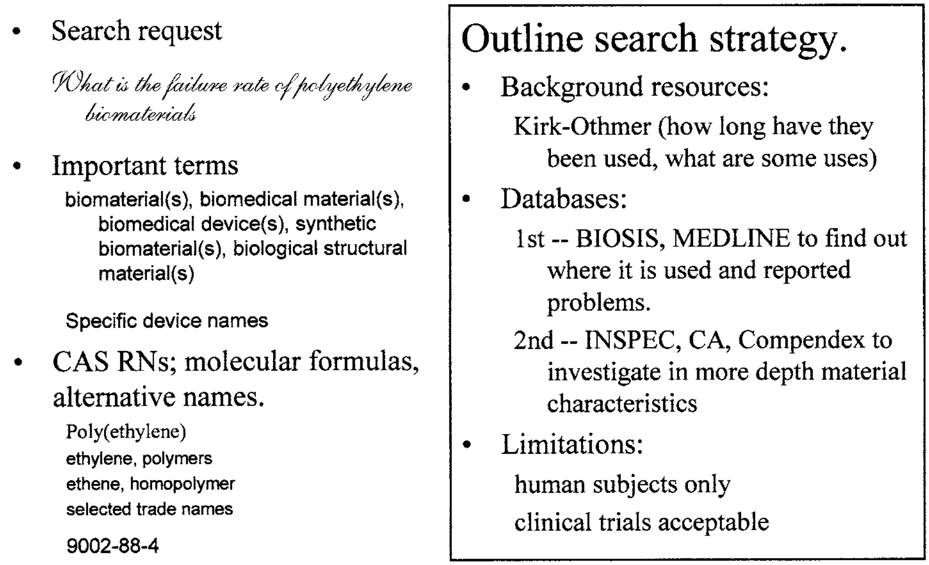
Question: What Is Thermal Conductivity of Diamonds?

Resource	Basic Information Available	Availability	Notes
Dictionary of Organic Compounds OR *Dictionary of Inorganic Compounds*	Synthesis, reaction, and property information, for organic and inorganic compounds of use to man; includes references	In the library with online index through the web	Quick browse in the online chemnetbase.com index reminds me that diamond is a material not a compound and not included in these type of resources.
Thermochemical Data of Organic Compounds	As stated in title	On my shelf	Won't work—diamond is a material compound
Kirk-Othmer Encyclopedia of Chemical Technology	Manufacturing and properties of chemical materials	Online version paid for by company and accessible from by database	Some data on diamond
Wiley Encyclopedia of Electrical and Electronics Engineering	Useful data and extensive sections detailing methods, etc.	Available online	Good resource for background and industry information
NBS Tables of Chemical Thermodynamic Properties, Donald Wagman, et al., NBS, 1982	Selected chemical thermodynamic properties	In the company library—a supplement to *Journal of Physical and Chemical Reference Data* OR NIST online files freely accessible from my desktop	This specific volume won't do as thermal conductivity is not included, but other tables might be useful. (In fact several of the other resources refer to Volume 1 of *The Journal of Physical and Chemical Reference Data*
CRC Handbook of Chemistry and Physics	Lots of data, references to original literature	On my shelf	Look up diamond or thermal conductivity, find tables, find references to more detailed data
Handbook of Physical Quantities by Igor S. Grigoriev and Evgenii Z. Meilikhov, ed., CRC Press, 1997	Detailed information on physical properties including explanations of methods and calculations, references to more detailed sources	In the company library	Has a nice table on thermal conductivity of elements. Diamond is listed and there are references to four more detailed sources
Rock Physics and Phase Relations, Thomas J. Ahrens, ed., American Geophysical Union, 1995	Physical properties of rocks and minerals	In the company library	Not needed today, but something to keep in mind—going outside the traditional when the traditional resources don't work
Landolt Zahlenwerte und Funktionen aus Naturwissenschaften und Technik, neue Serie by Gesamtherausgabe, K.H. Hellwege, Springer Verlag, 1961–	Extensive data on properties of materials	At the local university library	Difficult to use, will ask the librarian if more data is needed

reasonable time spans and cost budgets. I will add some comments on how to work with information professionals effectively and some of the electronic resources that are available to help you keep current.

2 THE PROCESS

Defining the Question. What is it you really want to find? Too often the materials engineer limits his or her answer set by asking for what she or he thinks can be found rather than what he or she really wants and needs. Often this is due to time restrains because the information or data located in less used information sources are not as immediately understood or as quickly incorporated into projects as those found in more familiar resources. These issues need to be addressed up front in order to get the information you need as quickly as you need it. Figure 2 shows a simple question or search form to help materials engineers define their questions clearly and prepare for the information hunt. The *search request* is a sentence or short paragraph that clearly states what information you need. It helps the engineer to take a moment and write out the search request. This clarifies what information is needed as well as what information is already known. In addition, a written search request makes it easier to communicate with information professionals when their help is needed. *Important terms* are those words that will be used to find information in indexes and with search engines. This form is not static; additional terms should be added as the search for data progresses. Different tools use different terminology to refer to the same or similar topics. Also, different fields of study may use the same term differently. Finally, terminology and how it is applied changes over the years. In most databases, the indexing is not updated, so the searcher needs to be familiar with all possible indexing choices. Here are some examples of these issues.



- Search request

  What is the failure rate of polyethylene biomaterials

- Important terms

  biomaterial(s), biomedical material(s), biomedical device(s), synthetic biomaterial(s), biological structural material(s)

  Specific device names

- CAS RNs; molecular formulas, alternative names.

  Poly(ethylene)
  ethylene, polymers
  ethene, homopolymer
  selected trade names

  9002-88-4

Outline search strategy.

- Background resources:

  Kirk-Othmer (how long have they been used, what are some uses)

- Databases:

  1st -- BIOSIS, MEDLINE to find out where it is used and reported problems.

  2nd -- INSPEC, CA, Compendex to investigate in more depth material characteristics

- Limitations:

  human subjects only

  clinical trials acceptable

Fig. 2 Searching worksheet.

Example 1: Spelling Variations

Recently, a scientist wanted information about "eschelle spectroscopy." When only nine abstracts were obtained with that phrase, I suggested we look at the spelling of "eschelle." Two variant spellings were in one of the nine abstracts retrieved. "Eschelle" was in the title and "echelle" was in the indexing. With the alternative spelling over 100 citations were found allowing further specification to include ICP (inductively coupled plasma) as a technique. Twenty-one citations were found with very specific information relevant to the work being done.

Over the years, these types of instances have shown me to always think broadly when dealing with terminology or important information will be missed, especially in cross-disciplinary searches. In the print world, these spellings might have been relatively close to each other in the index, and serendipity would have played its role in finding information. In the online world the searcher has to think broadly enough to make up for this loss of serendipity. Alternative spellings, abbreviations, and acronyms should always be included in a search in order to retrieve good resources.

Example 2: Terminology Changes over Time and Emphasis Varies by Discipline

Biomaterials is a cross-disciplinary area of research. To be effective in developing materials, the materials engineer may need to refer to the medical literature, chemical databases, and biological resources as well as the more traditional engineering resources. How many resources might be needed will vary depending on the nature of the search and how comprehensive the results need to be. Beginning investigation may not need multiple sources or databases. Dealing with a process or material that may eventually need to be patented will definitely require searching in multiple sources to find prior art.

The concept of biomaterials seems to be well defined in the engineering community today. *The Biomedical Engineering Handbook* states: "A biomaterial is used to make devices to replace a part or a function of the body in a safe, reliable, economic, and physiologically acceptable manner."[1] Another current definition of biomaterials is: "An engineering material suitable for use in situations where it may come into direct contact with the internal tissues of the body. Sometimes, biomaterials is used to refer to biological structural materials."[2] Notice the difference in emphasis on how the material will be used. Biomaterials have been in use since the 1800s. As late as the 1970s biomaterials were more often referred to under the heading of biocompatible materials.[3] "Synthetic biomaterials can be referred to as biomedical materials to distinguish them from naturally occurring biomaterials."[4]

The medical community refers to biomaterials as "any substance (other than a drug) synthetic or natural, that can be used as a system or part of a system that treats, augments, or replaces any tissue, organ or function of the body."[5] Currently the medical community tends to use specific names for types of biomaterials as well as the applications of these materials rather than the general term biomaterials.

The chemical community, as usual, focuses on the compound or class of materials such as polymers, ceramics, and metals with biomaterials or biomedical materials being considered a use rather than a type of material.

All of these differences need to be taken into consideration as the literature is searched. In this example the following terms would be included as possible search points: biomaterial(s), biomedical material(s), biomedical device(s), synthetic biomaterial(s), biological structural material(s), as well as more specific terms and chemical names relating to the specific interest.

One area where differences in terminology are definitely a factor is in chemical names. This means it is a good idea to identify terms like the Chemical Abstracts Service Registry Number (CAS RN), common names, trade names, and IUPAC or CAS names. Which of these names you know will often help you choose what tool to use to find the information or data you need. Again, variations on the chemical names will need to be added as the search progresses. Polymers is one area where nomenclature is very tricky, partly due to the large number of trade names as well as to seemingly insignificant changes in the chemical makeup of the polymer.

Example 3: Chemical Compounds and Chemical Classes

Polyethylene is listed as a nonaromatic hydrocarbon backbone polymer in *the Physical Properties of Polymer Handbook.*[6] A slight name variation is noticed in the online edition of the *CRC Handbook of Chemistry and Physics*—poly(ethylene)—with a broader class name given as polyalkenes.[7] These differences are not critical in the paper world but might be in an electronic database. *Chemical Abstracts* has called this material by several names over the years. In the 8th Collective Period it was referred to as "ethylene, polymers." Starting with the 9th Collective Period it has been referred to as "ethene, homopolymer." The registry number 9002-88-4 has over 4500 alternative names (many trade names). There are also 84 deleted registry numbers (or compounds that at one time were considered unique and have since been reviewed and found to be "ethene, homopolymer.") There are at least 49 files available on STN International (a vendor of databases with scientific bibliographic and numeric information; more fully described at the end of the chapter), a searching system that has information about the compound with registry number 9002-88-4. Each of these files has different methods of inputting chemical and material names, and each will provide different types of information. BIOSIS (the online database devoted to biological literature, described in depth later in the chapter), when searching for "polyethylene" as a controlled term, has 997 (search done on 4/28/01) citations. However, on the STN system, registry numbers have been added to the BIOSIS indexing for chemical compounds listed in the title of the article. Using the registry number as a search term yielded 2706 additional citations. When these citations were reviewed, it became quickly obvious that trade names were often used to refer to the polymer in the BIOSIS file. Without searching the registry number, important information would have been missed and the materials engineer might have missed, an important possible use for the material being studied or the fact that some other company has already gotten approval for clinical trials with a product. Missing information in either case will cost the company.

Locating Needed Information. Once the research area has been well defined, it is time to move onto the *search strategy*. There are three major decisions that need to be made. First, is background information needed? If so, what are the background resources that should be consulted to gather basic information relating to the project? If background information is not needed, it is possible that the appropriate background resources will provide leads as to what databases should be investigated.

Second, what databases (defined as a searchable source of information in paper or electronic format including data compilations, handbooks, and literature resources) will be appropriate to find the information you need. Do not limit the research to known databases in engineering. Materials science is cross disciplinary in nature. Literature databases in many other fields such as biology (BIOSIS), physics (INSPEC), medicine (MEDLINE) and chemistry (*Chemical Abstracts*) as well as Engineering Index (COMPENDEX) should all be consulted. The number of databases that need to be accessed depends on how comprehensive the information needs to be. These databases are discussed in more detail later in this chapter. Materials that may end up in a patent need to be researched in great depth using both print and online databases. Special attention to patent resource files such as Derwent and the U.S. Patent files will be needed when preparing for patent processes.

When dealing with data sources (handbooks, electronic databases, etc.), it is also wise to consider not only those that are well know in materials science but also those in related disciplines. Though the data needed may be in different formats or measured in different manners, they can still be useful when designing new materials. Chapter 16 discusses data sources and search strategies that vary depending on the type of data needed in considerable depth.

Third, are there any limitations that need to be considered before going into the resources? Often researchers provide themselves with false limitations such as:

> If the data is not available in an electronic format, then it doesn't exist.
>
> Anything published before 1960 is not accurate.

One example is the vast amount of information available through Gmelin, short for *Gmelins Handbuch der Anorganischen Chemie*, which contains data on inorganic compounds and materials collected and published from the 1800s. Though the data are old, and further testing may be needed with today's analytical tools, the data are well researched in considerable depth and are sometimes the only data available. This tool has been made easier to use with the advent of Crossfire, which provides an extensive index with some of the property information in an electronic format. This tool may provide helpful starting points for more in-depth investigation. For example, Gmelin has six volumes dealing with molybdenum and compounds of molybdenum. Additional information can be found in other volumes as well. The 4-page section in the main volume (published in 1935) detailing the thermal properties of elemental Mo included data on the specific heat measured from −253 to 1500°C. Referenced data for these results go back to *Annales der Chimie et de Physique* in 1840. The Gmelin database, as loaded on STN, cites 44 measurements of heat capacity at a constant pressure and 10 measurements of heat capacity at a constant volume. One of the later measurements comes from *Physical Review B,* vol 44, 1991. Over 150 years of literature is covered in this database.

Limitations that will be helpful include project deadlines, details about the physical state of the materials being researched, material characteristics and physical properties that are critical to the project goals, and cost limitations. Deadlines and cost limitations become very important when the information specialist is consulted. Generally speaking the sooner the deadline the more expensive the search process will be.

Using Best Known Resources First. Taking the time to be open to different possibilities means the results of the process may be very different than the expected "correct answer" foreseen at the beginning of the process. Also, there are a vast number of data resources to be considered, each with it own quirks. A busy materials engineer does not have the time to learn how to use them all. Take a look at Chapter 16 to get an idea of the many databases and information sources available. The best and most knowledgeable materials engineer cannot possibly know more than a few of these resources well. However, now that many handbooks have electronic versions and databases are developing more user-friendly search interfaces, it is possible to have access at your desktop to many familiar handbooks and databases. Searching for clues to information not as readily available can be done without leaving your office by using both paper and electronic resources.

Each materials engineer should develop his or her own desktop library. This collection will be a combination of print and electronic resources with which the materials engineer is familiar. Electronic versions of familiar resources should be used when the electronic interface adds to the value of the resource or space does not allow for the printed item. The desktop library should be considered both a fast answer resource as well as a place to find more clues to solve the physical property logic puzzle. The desktop library should contain at least one major handbook as well as some specialized handbook or data compilations. How many of these resources are used will depend on the space and funds available.

With the advent of online encyclopedias, the Desktop Library can contain a good resource for background information. Look at the electronic version of *Ullmann's Encyclopedia of Industrial Chemistry* and *Kirk-Othmer Encyclopedia of Chemical Technology* as possible resources. Access to the primary literature can be gained through full-text journals with or without use of indexing and abstracting tools. Two useful journals, now available full text are *Journal of Physical and Chemical Reference Data* (http://ojps.aip.org/jpcrd/) and *Journal of Chemical & Engineering Data* (http://pubs.acs.org/journals/jceaax/index.html). Both of these titles have been terrific data sources for many years.

Three hard copy handbooks (which can also be accessed electronically) that provide both basic information and leads to more extensive resources come to mind—*Perry's Chemical Engineers' Handbook, Engineering Handbook* by Dorf, and *CRC Handbook of Chemistry and Physics.* Several materials-related handbooks are also useful resources—*Materials Handbook* by Brady and Clauser and the *CRC Materials Science and Engineering Handbook.* These are only a few of the titles available. The materials engineer should find the handbook(s) that fits his or her needs best. Once the more general handbooks have been selected, more specific subject/project-related handbooks are helpful. For example, an excellent resource in physical property information for polymer science is the *Physical Properties of Polymers Handbook* by James E. Mark, published by the American Institute of Physics. Notice that the handbooks mentioned are from a variety of disciplines that might be of use to a materials engineer.

The resources chosen should include basic information needed on a regular basis as well as charts and tables of information that might prove useful. For example, the *CRC Handbook of Chemistry and Physics* has very familiar tables of basic property information for organic and inorganic materials. But, unused by many, is the wealth of tables on many topics of interest to the materials engineer. I have already mentioned the Glass Transition Temperature for Selected Polymers. This table, by Robert B. Fox, has properties that were determined for the purpose of generating this table. Many tables are compilations of data from several sources, and the editors of the handbook are careful to note what resources were used. A good example of this is another table in the same section—Dielectric Constant of Selected Polymers. This table references three sources:

1. Gray, D. E., Ed., *American Institute of Physics Handbook,* 3rd ed., McGraw-Hill, New York, 1972, p. 5–132.
2. Anderson, H. L., Ed., *A Physicist's Desk Reference,* American Institute of Physics, New York, 1989.
3. Brandrup, J., and Immergut, E. H., *Polymer Handbook,* 3rd ed., Wiley, New York, 1989.

This reference list provides several pieces of information. First, it provides references to sources that may have more detailed information than that provided in this table (including the analytical methods used to generate the data). Second, they state the approximate "age" of the information. Because the dates are all prior to 1989, it becomes probable that additional information is available in the primary literature, in more current handbooks such as the *Physical Properties of Polymers Handbook* mentioned earlier, or in later editions of the handbooks referenced. Third, the references provide you with evaluative information. Knowing that the data has been in the published literature adds additional credence similar to peer review for other types of scientific literature. This is critical, as many online web pages with physical data on various compounds and materials do not tell you how the information is obtained, what the experimental conditions are, or even the purity of the material being tested. Starting with the known resource such as a favorite handbook, a physical property database, or a full-text journal, can lead to quicker results and very complete answers. With so many handbooks and data compilations available at the desktop, it is important for the materials engineer to make the following decisions on what to include based on the following recommendations:

Include materials in your desktop library that you will use often. Know how to use your desktop library well.

Include items that have the type data or information needed routinely. Find out what is included in each resource as well as what limits (i.e., only data from patents is included) have been placed on inclusion.

Include items where access methods, such as indexing, table of contents, and online search options, are easy to use.

Include authoritative resources with good literature references and clearly noted experimental conditions.

Going beyond Desktop. Once the familiar resources have been exhausted, it is time to consider outside help. Obviously, if you found the information you need and you are relatively certain you do not need additional verification, the search can stop at any time. However, if the desktop library has not led to the information you need or the project is being considered for publication or patenting, it is time to see the information expert in your organization.

The structure for getting information varies in each organization. It is important to understand the information climate or your organization. Some expect the materials engineer to find it all on their own. Some feel looking for information is not an effective use of the materials engineer's time and expects all such research efforts to be turned over to an information specialist. Most organizations have information centers, libraries, or reading rooms. Smaller companies or colleges may have limited access to information resources and external libraries or information brokers must be used. Many of the larger organizations will have information specialists or librarians. Smaller organizations may not have specialists in the area you are working, however, information professionals are all specialists in data organization and retrieval. So, even if they do not have the subject expertise you would like, they will often be able to help you use the tools effectively.

When to use these resources will depend on the information need. Find out what is preferred in your organization and do not limit your exploration to what the folks in your direct department do unless it is the best way for you to find the information you need. Often there are resources outside the department that will also help. In some places use of these external resources are not encouraged as there is a cost attached. In that case, try to weigh what the cost will be if the information needed is not found—e.g., millions of dollars spent on developing a product that someone else patented 2 years ago—against the cost of a good information search.

Many standard resources are now available to the engineer electronically and can be incorporated into the desktop library discussed previously. A cautionary note here. Make sure proper training on the use of even the basic resources is taken. As mentioned before, even the simplest of resources could have a wealth of information an engineer is not aware of because they have not used it to its fullest capacity. A tool you have used throughout your professional life may have more information than you thought or may be used in different ways for better results. As electronic resources become more prevalent, the method of use may change from year to year, and a resource you knew how to use last month may have a new interface this month. The information professional is the expert on how to use information products. Information specialists and librarians generally enjoy showing the researcher and engineer how to use resources to their fullest capabilities. Many organizations will have routine training opportunities for those resources that are of the most use to their employees. Taking the time to learn these resources well will not only save you considerable amount of time later but the training opportunities will give you a chance to get to know the information staff at the organization. Then, when you need their help, they may already know what you are working on. This can speed up the process considerably. One additional note, some engineers are not comfortable with sharing their ideas at an early stage. Be assured information professionals know that confidentiality is an important part of their work. Your queries will not be shared with others.

Find out who the information gatekeeper is within your immediate organization. Sometimes there is a person who enjoys working with information who can guide you to the best resources and the best information specialist or librarian for your specific needs. In smaller organizations the information gatekeeper may indeed be the only information specialist available.

Always make use of internal resources first. Use the corporate library or information center as the first stop after exhausting your desktop library and your department's information gatekeeper. **Do not make the web your first stop outside your department!** This does not mean to ignore valuable resources that are available electronically through the Internet, such as the NIST databases and the many fine reference tools available. Rather it means use known resources, including your organization library and librarians (information center and information specialists), and purchased and attributed data sources accessed through the Internet before searching the highly disorganized web. The web contains vast amounts of information. However much of it has no attribution and little of it is organized so that information can be easily found. Anyone can post a page to the web for any reason. No quality check required. One excellent Internet resource is product information available in fact sheets and material safety data sheets (MSDS). As these are part of how a company markets its items, they should contain reliable information. At my institution, I tell students that when it is time to use the web, start at the library's home page where the librarians have already sorted through the web resources available and found some of the better ones to recommend. Also, the home page is where links are made to those electronic resources with web interfaces that the library purchases for students and faculty use.

After exhausting internal resources, find out what external organizations your company uses at a fee to get the information you need. If your organization has a library or information center, this may be transparent, i.e., the information center will provide the information to you by going to an outside organization. These services are called InterLibrary Loan (ILL) or document delivery by the library world. If your organization does not have such a service, it may have purchased information research skills through information consultants or brokers. If it has done so, these consultants are most likely aware of the general needs for projects within your organization and may have specialized training in retrieving information applicable to your needs.

It is a good idea to determine what local colleges or universities have agreements with your organization that will allow you to use their collections. Unless your have a large library, browsing through a university research collection can be a fruitful way to gather information. For a small organization without internal information resources, local academic libraries are critical.

Academic libraries are the storehouse of information for the research community. However, this does not mean this information is accessible to every one. Public colleges and universities usually have some mandate to provide access to their collections and perhaps some services to residents of the state. What the limits of this access are will vary. Private academic communities do not have any obligation to provide such access, although many do. In either case, access methods and procedures will vary:

1. Check with the company library to find out if access to collections has been purchased. Academic libraries may be willing to allow members of local organizations to check out materials from their library upon purchase of a library card. Also, many companies do have ILL agreements that allow the company library to request copies of materials.
2. See if the reference services are available to noncommunity members. Generally a limited amount of reference service is available to the public, but this is not always the case. Reference staff like to help people find information. Being polite and friendly, asking interesting questions, understanding you are a guest, using services during slow times of the academic year, as well as being from an organization that supports the university can all mean your reference queries are taken seriously.
3. Determine what resources are accessible by noncommunity members. It used to be that anyone allowed in the door could use the paper copy *Engineering Index.* Now that the tool is in electronic format there may be restrictions on use for noncommunity members due to licensing agreements or searching costs.
4. Some academic libraries offer a fee for service arrangement. These can be very important to small companies. The costs are usually set up to recover any out-of-pocket expenses as well as some amount for the librarians' time.
5. Be aware that collections, electronic resources, and services cost money. If your company is relying on a local academic library, remember to offer assistance financially. This can often be done through Friends of the Library organizations, paying for research services, or by providing research funding (though this route will not get funding directly to the library).

Evaluating Data/Information Resource. One of the themes running through this chapter has been quality of information resources. This quality is seen by both the way the resources are organized and the value and quality of information or data retrieved from the resources. Knowing how to evaluate information for quality is critical. It is important because bad data/information leads to poorly designed materials and bad products. For example, the shuttle *Challenger* disaster could have been avoided if better data on the stress and temperature factors affect the rubber rings had been incorporated into the design specifications.

There are several ways to evaluate the information you find. First, look at the quality of the resource being used. Handbooks that have been around for many editions and printings will generally have time-tested information. Property data from a well-know handbook is always better than a value found on some web page without any attribution. Peer-reviewed journal articles may provide better data than trade publications. Second, data that has experimental parameters defined is always better than "naked" information. The more known about how the information was collected the better. Third, if the person preparing the data source has not provided information on how it was collected or who measured it, it should only be used with caution. Fourth, use caution with information obtained from web pages. Remember that anyone can put up a web page. There is no requirement that data on a web page be accurate. Check the page out. Is there an author specified? Was the information put up by an unbiased organization? If the page is from a university, is the author a researcher or a student doing a class project? If the information is from a company, is it trying to sell you something?

It is the materials engineer's responsibility to evaluate the information. The best scenario is to have multiple sources of information to quality check. This is rarely possible, and, sometime when it is, further checking many find that the data from multiple secondary resources came from the same primary resource. So the data and information must always be used within context and with care. Sometimes that means starting again because the data or information is suspect.

Redefining the Question Using New Information. Starting again does not mean starting over. Usually there has been some information gathered that allows the materials engineer to refine the search question either more broadly or more narrowly. If no data has been found, broaden the topic and see if related information can be used. Can other properties be used to provide information if the specific one needed is not reported? When too much information has been found, narrow the topic wisely. Again, do not assume you want to narrow the scope by using only familiar resources. Sometimes the narrowing needs to be done by using resources outside the field that specialize in the information needed. Remember information specialists and librarians are experts in refining the search question.

Knowing When Information Gathered Is Enough. Unlike a logic puzzle, data research does not always have an answer. Sometimes there is no data. For the researcher trying to delve into a new area, this might be wonderful. For the materials engineer trying to modify an existing material, this can be annoying. However, after you have broadened the search, after you have found related information but not exactly what you want, and after an information professional states the information does not appear to be the *published* literature, then it is time to call on colleagues (if you have not before). Then searching the web might bring up a new lead. Or then it may simply be time to stop and go with what you have. Only the materials engineer can make that decision.

3 DATABASES

Big Five Literature Databases. All of the major science literature databases provide indexing and abstracting of information from the primary literature from around the world. All contain full bibliographic information that is retrieved through subject indexing and free text searching of titles and abstracts. Each database contains some references to non-English language materials. The types of primary literature covered vary by discipline. All of the databases are available through major database vendors such as DIALOG, STN International, OCLC FirstSearch, Axiom (from IOP), and OVID. Many also have front ends and web search engines direct from the publisher. Different search features are available through different search interfaces. Most databases include title, abstract, indexing, and references. Many provide links to full-text information. The most up-to-date information about access options can be found on the databases or database vendors home pages. One way to evaluate which database to use is to look at the list of titles covered in a particular database. Note which titles are in a database "cover to cover" (i.e., all articles are entered) and which are selectively added.

1. *BIOSIS—Biological Abstracts.* BIOSIS, Two Commerce Square, 2001 Market Street, Suite 700, Philadelphia, PA 19103-7095. Phone: 1–800–523–4806 or +1–215–587–4800. Fax: +1–215–587–2016. Web: http://www.biosis.org. E-mail: info@mail.biosis.org . Indexes and abstracts in the biological, biochemical, and life sciences articles, books, dissertations, and conference proceedings. It has an extensive biological classification scheme. Available through most major database vendors. An added feature through STN: addition of CAS registry numbers to the indexes for compound listed in the article title. An excellent resource for biomaterials.

2. *INSPEC—Physics Abstracts, Electrical and Electronics Abstracts, Computer and Control Abstracts.* Institute for Electrical Engineers, Michael Faraday House, Six Hills Way, Stevenage, Herts, SG1 2AY, United Kingdom. Phone: (+44) 1438/313311, US 732-321-5575. Fax: (+44) 1438/742840, US 732-321-5702. E-mail: inspec@iee.org.uk, or inspec@inspecinc.com. 1969 to present. International in scope, the database contains over 2 million records for articles, conference proceedings, and books. The materials engineer should use this database to access information, pertaining to the physical properties of materials, in areas of physics, polymers, ceramics, computational methods, and biophysics. An interesting feature of this database is the element and physical property fields that can allow the materials engineer to specify what type of chemical components are discussed in the primary literature. An excellent resource for information on sold-state materials.

3. *MEDLINE—Index Medicus.* National Library of Medicine, MEDLARS Management Section, 8600 Rockville Pike, Bethesda, MD 20894. Phone: 1–800–638–8480. Fax: +1–301–496–0822. Web: http://www.nlm.nih.gov. E-mail: mms@nlm.nih.gov. Indexes and abstracts the fields of medicine, nursing, dentistry, veterinary medicine, the health care system, and the preclinical sciences. MEDLINE contains bibliographic citations and author abstracts from more than 4000 biomedical journals published in the United States and 70 other countries. Available through many database vendors and directly (at no cost) from the National Library of Medicine. PubMed also provides access and links to the integrated molecular biology databases included in NCBI's Entrez retrieval system. These databases contain DNA and protein sequences, 3-D protein structure data, population study data sets, and assemblies of complete genomes in an integrated system.[8]

4. *CAS—Chemical Abstracts and the Registry File.* Chemical Abstract Service, 2540 Olentangy River Road. Columbus, Ohio 43202. Phone: 1–614–447–3700. Fax: 1–614–447–3767. Web: http://www.cas.org. Email: help@cas.org. CAS indexes and abstracts in chemistry, biochemistry, and other chemical sciences papers, books, conference proceedings, patents, technical reports, and dissertations worldwide (1907 to current). Chemical name, chemical structure, genome, and protein sequence searching are available in the Registry. Other CAS files provide detailed synthesis and reaction information. For ceramics, inorganic materials, and organometalic materials searching molecular formula, element counts and ring systems in the Registry file allow a high level of specificity as well as broad class identification of compounds and materials. The most complete versions of these files is available through STN International; however, the basic databases are available through all major database vendors.

5. *COMPENDEX (or EI or Engineering Index).* Engineering Information Inc., EI Headquarters, 1 Castle Point Terrace, Hoboken, NJ 07030-5996. Tel: (+1) 201–216–8500. Fax: (+1) 201–216–8532. Toll free: (+1) 800–221–1044 US and Canada. Web: http://www.ei.org/. E-mail: ei@ei.org. Indexes and abstracts information from the engineering and technological literature including over 2600 international journals, conference papers, and technical reports. Of special interest to materials engineers is the chemical, biomedical, and metals formation.

Other Databases

1. *Science Citation Index (part of Web of Science).* ISI 3501, Market Street, Philadelphia, PA 19104. Phone: 800–336–4474 or 215–386–0100. Fax: 215–386–6362. Web: http://www.isinet.com/isi/. A citation index provides ready access to information on what papers are routinely being cited. They provide a method to broaden searches by finding out how seminal papers have been cited. Several database producers are now adding citation searching within their subject areas.

Science Citation Index provides access to current and retrospective bibliographic information, author abstracts, and cited references found in 3500 of the world's leading scholarly science and technical journals covering more than 150 disciplines. Its focus is on the most heavily used materials in all of science, thus allowing cross-disciplinary searching and retrieval of the most important references. (A related product Materials Science Citation Index may also be of interest as it provides a more focused group of journals.) Keyword and title word searching is available, making this a useful but limited tool for subject searching. This database is best used in conjunction with more in-depth and formally indexed subject database.

2. *ProceedingsFirst.* OCLC Online Computer Library Center, Inc., 6565 Frantz Road, Dublin, Ohio 43017-3395. Phone: 1–800–848–5878. Fax: +1–614–764–6096. Web: http://www.oclc.org. E-mail: support@oclc.org. Provides citations of every congress, symposium, conference, exposition, workshop, and meeting received at The British Library. Contains in each record a list of the papers presented at each conference.

3. *Derwent World Patents Index.* Derwent Information, 1725 Duke Street, Suite 250, Alexandria, VA 22314. Telephone: + 1–703–706–4220. Fax: +1–800–457–0850. Web: http://www.derwent.com/index.html. E-mail: custserv@derwentus.com. Derwent reviews and processes more than a million patents a year from over 40 international patent issuing authorities, as well as research papers and articles from more than 1200 other sources. The World Patents Index is one of the files they offer. Extensive chemical and engineering related indexing terminology as well as Markush structure searching is available in these resources.

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8. http://www.ncbi.nlm.nih.gov:80/entrez/query/static/overview.html#Database Coverage, May 1, 2001.

CHAPTER 16

SOURCES OF MATERIALS DATA

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1 INTRODUCTION AND SCOPE

It is the purpose of this chapter to aid engineers and materials scientists in locating reliable sources of high-quality materials property data. While sources in hardcopy form are referenced, the main focus is on electronic sources that provide well-documented searchable property data.

To identify useful sources of materials data, it is important to have clearly in mind at the outset (a) the intended use of data, (b) the type of data required, and (c) the quality of data required. These three factors are key in narrowing a search and improving its efficiency of a search for property data. Therefore, as an introduction to the identification of some specific potentially useful sources of materials data, we will discuss those three factors in some detail, and then describe the options available in types of data sources.

It is beyond the scope of this publication to attempt to provide a comprehensive list of all of the several thousand sources of materials data in various forms and formats. Readers interested in a more comprehensive list of sources and of more discussion of the technology of material property data technology and terminology are referred to Westbrook's extensive treatment of these subjects in

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Handbook of Materials Selection. Edited by Myer Kutz

Refs. 1 and 2 and to *ASM International Directory of Material Property Databases.*[3]

2 INTENDED USES FOR DATA

Numeric material property data are typically needed for one of the following purposes by individuals performing the respective functions as part of their jobs:

- Modeling material or product performance
- Materials selection (finding candidate materials for specific applications)
- Analytical comparisons (narrowing the choices)
- Preliminary design (initially sizing the components)
- Final design (assuring performance; setting performance specifications)
- Material specification (defining specifications for purchase)
- Manufacturing (assuring processes to achieve desired product)
- Quality assurance (monitoring manufacturing quality)
- Maintenance (repairing deterioration/damage)
- Failure analysis (figuring out what went wrong)

It is useful to note some of the differing characteristics of data needed for these different functions.

2.1 Modeling Material and/or Product Performance

To an increasing extent, mathematic modeling is used to establish the first estimates of the required product performance and material behavior, and even in some cases the optimum manufacturing processes that should be used to achieve the desired performance. The processes and/or performance analyzed and represented may include any of the issues addressed in the following paragraphs, and so the nature of the types of data described under the various needs are the same as those needed for the modeling process itself.

2.2 Materials Selection

The needs of materials specialists and engineers looking for materials data to aid in the selection of a material for some specific application are likely to be influenced by whether they are (a) in the early stages of their process or (b) have already narrowed the options down to two or three candidates and are trying to make the final choice. The second situation is covered in Section 2.3, Analytical Comparisons.

If the materials engineers are in the early stages of finding candidate materials for the application, they are likely to be looking for a wide variety of properties for a number of candidate materials. More often, however, they may decide to focus on two or three key properties that most closely define the requirements for that application and search for all possible materials providing relatively favorable combinations of those key properties. In either case, they may not be as much concerned about the quality and statistical reliability of the data at this stage as much as the ability to find a wide variety of candidates and to make direct comparisons of the performance of those candidates.

In cases where there is interest in including relatively newly developed materials in the survey, it may be necessary to be satisfied with only a few representative test results or even educated "guesstimates" of how the new materials may be expected to perform. The engineers will need to be able to translate these few data into comparisons with the more established materials, but at this stage they are probably most concerned with not missing out on important new materials.

Thus, at this early stage of materials selection, the decision makers may be willing to accept data rather widely ranging in type and quality, with few restrictions on statistical reliability. They may even be satisfied with quite limited data to identify a candidate that may merit further evaluation.

2.3 Analytical Comparisons

If on the other hand, the task is to make a final decision on which of two or three candidate materials should be selected for design implementation (the process defined here as "analytical comparison"), the quality and reliability of the data become substantially more important, particularly with regard to the key performance requirements for the application. It will be important that all of those key properties, e.g., density, tensile yield strength, and plane-strain fracture toughness, are available for all of the candidate materials that may be the next cut in the list.

The search will also be for data sources where the background of the data are well defined in terms of the number of tests made, the number of different lots tested, and whether the numbers included in the data source are averages or the result of some statistical calculation such as that to define three σ limits, for example. It would not be appropriate at this stage to be uncertain whether the available data represent typical, average values or statistically minimum properties; it may not be important which they are, but the same quality and reliability must be available for all of the final candidates for a useful decision to be made.

In addition, the ability to make direct comparisons of properties generated by essentially the same, ideally standard, methods is very important. The decision maker will want to be able to determine if the properties reported were determined from the same or similar procedures and whether or not those procedures conformed to ASTM International, International Organization for Standardization (ISO),[4] or other applicable standard test methods.

One final requirement is added at this stage: The materials themselves for which the data are presented must all represent to the degree possible comparable stages of material production history. It would be unwise to base serious decisions on comparisons of data for a laboratory sample on one hand and a commercial size production lot on the other. Laboratory samples have a regrettable history of promising performance seldom replicated in production-size lots.

Thus, for analytical comparisons for final candidate material selection, specialists need databases for which a relatively complete background of metadata (i.e., data about the data) are included and readily accessible.

Incidentally, it is not unusual at this point in the total process to decide that more data are needed for a particular candidate than are available in any existing database, and so a new series of tests are needed to increase confidence in the comparisons being made.

2.4 Preliminary Design

Once a decision is made on a candidate material (or sometimes two) for an application, the task of designing a real component out of that material begins. The requirement for statistical reliability steps up, and the importance of the availability of a data source that provides applicable metadata covering quality, reliability, and material history becomes even more important.

At this stage, the statistical reliability required includes not only a minimum value but one based upon a statistically significant sample size, ideally something comparable to the standards required in the establishment of MIL-HDBK-5's A or B values.[5] In MIL-HDBK-5 terminology, an A value is one that would be expected to be equaled or exceeded by 99% of the lots tested with 95% confidence; the B value provides for 90% of lots tested equaling or exceeding the value with 95% confidence. Furthermore, the MIL-HDBK-5 guidelines require that A and B values be based upon predefined sample sizes, representing a minimum number of lots (normally 100 or more) and compositions (normally at least 3) of a given alloy. The provision of such statistical levels needs to be a part of data sources used for design purposes, and the description of the statistical quality needs to be readily available in the data source.

For preliminary design, then, the data sources sought will include both statistically reliable data and well-defined metadata concerning the quality and reliability provided.

2.5 Final Design

Setting the final design parameters for any component or structure typically requires not only data of the highest level of statistically reliability but also, in many cases, data that have been sanctioned by some group of experts for use for the given purpose. It is also not unusual that at this stage the need is identified for additional test data generated under conditions as close as possible to the intended service conditions, conditions perhaps not available from any commercial database.

Databases providing the level of information required at this stage often contain what are characterized as "evaluated" or "certified" values. Evaluated data are those that, in addition to whatever analytical or statistical treatment they have been given, have been overviewed by an expert or group of experts who make a judgment as to whether or not the data adequately and completely represent the intended service conditions, and if necessary incorporate their own analysis into the final figures. This technique has been widely used in digesting and promulgating representative physical property data for many years; examples are the thermophysical property data provided by Thermophysical Properties Research Center (TPRC).[6]

Other databases may be said to provide "certified" data. In this case, the database or set of data going into a database have been evaluated by a group of experts and certified as the appropriate ones to be used for the design of a particular type of structure. Two examples of this are the aforementioned MIL-HDBK-5 values,[5] which are approved for aircraft design by the MIL-HDBK-5

Coordination Committee consisting of aerospace materials experts; and the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code,[7] with properties certified by materials experts in that field for the design of pressure vessels and companion equipment for high-temperature chemical processes.

As noted, it is often the case when designers reach this stage (if not the earlier preliminary design stage) that they find it necessary to conduct additional tests of some very specialized type to ensure adequate performance under the specific conditions the component or structure will see in service but for which reliable databases have not previously been identified or developed. The net result is the creation of new materials databases to meet highly specialized needs in a manner that provides the appropriate level of statistical confidence.

2.6 Material Specification

Material specifications typically include specific property values that must be equaled or exceeded in tests of those materials that are being bought and sold. The properties that one requires in this case may differ from those needed for other purposes in two respects. First and foremost, they must be properties that will ensure that the material has been given the desired mechanical and thermal processing to consistently achieve the desired performance. The second requirement is that, while in most cases there may be only one or a very few properties required (most often tensile properties), they are required at a very high level of precision and accuracy, similar to or better than that required for the MIL-HDBK-5 A properties defined earlier.

Examples are the material specifications required for the purchase of commercial aluminum alloy products.[8] These are usually only the chemical composition and the tensile properties. So while many of the other properties needed for design are not required as part of the purchase specification, those properties that are required are needed with very high reliability. In the case of aluminum alloys, the requirements for tensile strength, yield strength, and elongation are normally that 99% of lots produced must have properties that equal or exceed the published purchase specification values with 95% confidence, and they must have been defined from more that 100 different production lots from two or more producers.

In many cases, the databases needed to generate material specification properties are proprietary and are contained within individual companies or within the organizations that set industry specifications. However, the resultant statistically reliable specification properties are resident and more readily available in industry or ASTM material standards.[4,8]

2.7 Manufacturing

The properties required for manufacturing purposes may be the most difficult to find in commercially available databases because they typically involve the specialized treatments or processes utilized by specific producers or suppliers of the specific products in question. Sometimes these processes are proprietary and closely held for competitive advantage. An added complication is that once some semifabricated component (e.g., aircraft sheet) has been purchased, it will require forming to very tight tolerances or finishing at some relatively high temperature.

The fabricator may require data to enable the process to be carried through without otherwise damaging or changing the properties of the component, but may have difficulty representing the fabricating conditions in meaningful tests. That fabricator may well have to carry out its own tests and build the needed database to provide the desired assurance of quality, and to provide a source of information to which its own employees can refer to answer specific questions. Typically such databases never become commercially available, and new situations will require compiling new data sources.

Some processing data sources are available, of course. The Aluminum Association, Inc., for example, provides to all interested parties a data source defining standard solution heat treating, artificial aging, and annealing treatments for aluminum alloys that will assure the proper levels of properties will be obtained.[8] In some cases, ASTM and the American National Standards Institute (ANSI) material specifications will also contain such information.

2.8 Quality Assurance

Quality assurance may be considered to be the flip side of material specifications, and so the types of data and the data sources themselves required for the two functions are essentially the same.

Purchasers of materials, for example, may chose to do their own testing of the materials once delivered to their facilities by materials producers. If so, they will use exactly the same tests and refer to exactly the same data sources to determine compliance. The one difference may be that such purchasers may chose to gradually accumulate the results of such tests and build their own databases for internal use, not only by their quality control experts, but also by their designers and materials experts who must establish safe levels of performance of the structures. These types of databases also tend to be proprietary, of course, and are seldom made available to the outside world, especially competitors.

2.9 Maintenance

The principal value of material data in connection with maintenance concerns will likely be for reference purposes when problems show up with either deterioration of surface conditions or the suspicion of the development of fatigue cracks at local stress raisers.

In both cases, the important features of the types of data desired to address such issues are more likely to be those based upon individual exposures or prior service experience, and so the user may be more concerned about the degree of applicability than upon data quality and statistical reliability (though both features would be desired if available). Typically such data are hard to find in any event, and once again are more likely to be buried in proprietary files than in published databases.

In the case of engineers and technicians needing databases comprised of service experience, they may well be faced with building their own data sources based upon their organization's production and service experience than expecting to be able to locate applicable external sources.

2.10 Failure Analysis

The occurrence of unexpected failure of components in a structure usually calls for some follow-up study to determine the cause and possible ways to avoid further loss. In such cases it is inevitable that such failure analysis will involve both (a) a review of the old databases used to design the part and (b) a search for or the development of new data sources that may shed more light on the material's response to conditions that developed during the life of the structure that had not been anticipated beforehand.

The types of databases sought in this case will likely be those containing statistically reliable data, but recognizing the unexpected nature of some problems, an interest in a wider range of data sources and a willingness to consider a lower level of data quality may result. Databases for failure analysis studies may need to be wider in scope and to cover subjects such as corrosion that are not always easily treated by statistical means. In fact, sources covering failure experience may be the most valuable, though hardest to find because historically engineers and scientists do not publish much detail about their mistakes.

The net result is that when dealing with failure analysis, the search may be quite broad in terms of data quality, and the focus most likely will be more on applicability to the problem than on the quality and structure of the compilation. As in the case of maintenance engineers and technicians needing databases comprised of service experience, failure analysts may well be faced with building new data sources based upon their organization's production and service experience.

3 TYPES OF DATA

It is useful at this stage to note that there are several basic types of materials databases, i.e., databases containing significantly different types of information and, hence, different data formats. Note that this is different from the type of platform or presentation format (e.g., hardcopy, CD, online, etc.); these will be discussed in Section 6.

The two fundamental types of databases discussed here are *textual data* and *numeric data*. In fact, many databases represent a combination of both types, but there are some basic differences worth noting as in the next two paragraphs. The concept of metadata will also be described in more detail in this section.

3.1 Textual Data

The terminology *textual data* is generally applied to data entries that are purely alphabetic in nature with numbers used only as necessary to complete the thoughts. Textual databases are typically searched with alphabetical strings, e.g., by searching for all occurrences of a term such as *aluminum* or *metallurgy* or whatever subject is of interest.

The subjects of textual databases are predominantly bibliographies and abstracts of publications, but they may reflect other specific subjects such as textual descriptions of failures of components, for example. Bibliographic databases seldom reflect the final answers to whatever inquiry is in mind but rather references to sources where the answers may be found.

The majority of all databases in existence in any form (see Section 6) are textual in nature, even many of those purporting to be property databases. Searchers of such textual property databases are searching based upon strings of alphanumeric characters reflecting their interest, not on numeric values of the properties except as they are expressed as strings. This is quite different from the case for numeric property databases, as we shall see in the next section.

3.2 Numeric Databases

Databases classed as numeric (a) have data stored in them in numeric format and (b) are searched numerically. For example, numeric databases may be searched for all materials having a specific property equal to or greater than a certain value or within a certain specific range; this would not be possible in a textual database. To provide such searchability, almost all numeric databases are electronic in nature (see Section 4) though many hardcopy publications also contain extensive amounts of numeric data.

To accommodate numeric searching, the properties must be entered into a database digitally as numbers not simply as alphanumeric strings. And to be useful to material specialists and designers, they must have meaningful precision (i.e., numbers of significant figures) and units associated with each number. The numeric number of a property is of no value if it does not have the applicable unit(s) associated with it.

3.3 Metadata

The concept of metadata as ‘‘data about data’’ was introduced earlier, and it is appropriate at this stage to describe the concept and its importance in greater detail.[9] It is vital, especially for numeric databases and independent of their platform, to have ample background information on the numeric properties included in the database and closely associated with the individual properties.

Examples of metadata include:

- Original source of data (e.g., from tests at ABC laboratories)
- Test methods by which data were obtained (e.g., ASTM methods, size and type of specimen)
- Production history of the samples for which the data are applicable (e.g., annealed, heat treated, cold worked, special handling?)
- Exposure experienced by the samples tested, if any, prior to the test (e.g., held 1000 h at 500°F)
- Conditions under which the properties were determined (e.g., tested at 300°F, 50% humidity)
- Number of individual tests represented by and statistical precision of the values presented (e.g., individual test results; averages of *x* number of tests, statistical basis)
- Any subsequent evaluation or certification of the data by experts (e.g., ASME Boiler & Pressure Vessel Committee)

It should be clear that the value of information in any database, but especially in a numeric database, is greatly diminished by the absence of at least some and potentially all of these types of metadata. For example, a listing of the properties of an alloy is of no value at all if it is not clear at what temperature they were determined. It should be clear that metadata are an integral part of every material property database. Similar properties of a material are of virtually no value if it is not clear how they were mechanically and thermally processed before they were tested.

4 SUBJECTS OF DATA SOURCES

It is next useful to note some of the major categories of data available and illustrate the manner in which they are likely to be classified or structured. From this point on, the discussion will focus primarily upon numeric materials property data of interest to the engineer and scientist.

The total breadth of properties and characteristics may reasonably be illustrated by the following four categories:

- Fundamental (atomic level) properties
- Physical properties (atomic and macro/alloy levels)
- Mechanical properties (macro/alloy level)
- Application performance (macro/alloy or component levels)

To provide greater detail on the first three categories, it is helpful to utilize the taxonomy of materials information developed by Westbrook[1] and illustrated in Fig. 1. While the entire list of potential uses of data described above is not included, the taxonomy in Fig. 1 illustrates the variety of subject matter quite well.

The fourth major category identified above involves what is referred to as application performance but which in itself incorporates several individual areas:

- Fabrication characteristics (sometimes called the “ilities”)
  - **(a)** Fabricability (“workability”)
  - **(b)** Forming characteristics (“formability”)
  - **(c)** Joining characteristics (“joinability”)
  - **(d)** Finishing characteristics (“finishability”)
- Service experience
  - **(a)** Exposure conditions
  - **(b)** Service history
  - **(c)** Failures observed and their causes

It is not necessary to discuss these categories individually, simply to recognize that specific databases may well focus on only one or a few of these subjects and that rarely if ever would all subjects be included in any one database.

5 DATA QUALITY AND RELIABILITY

It was noted in Section 2 that individuals doing preliminary material selection or screening may have different needs with regard to quality and reliability than those doing design functions. It is appropriate at this stage to review the major

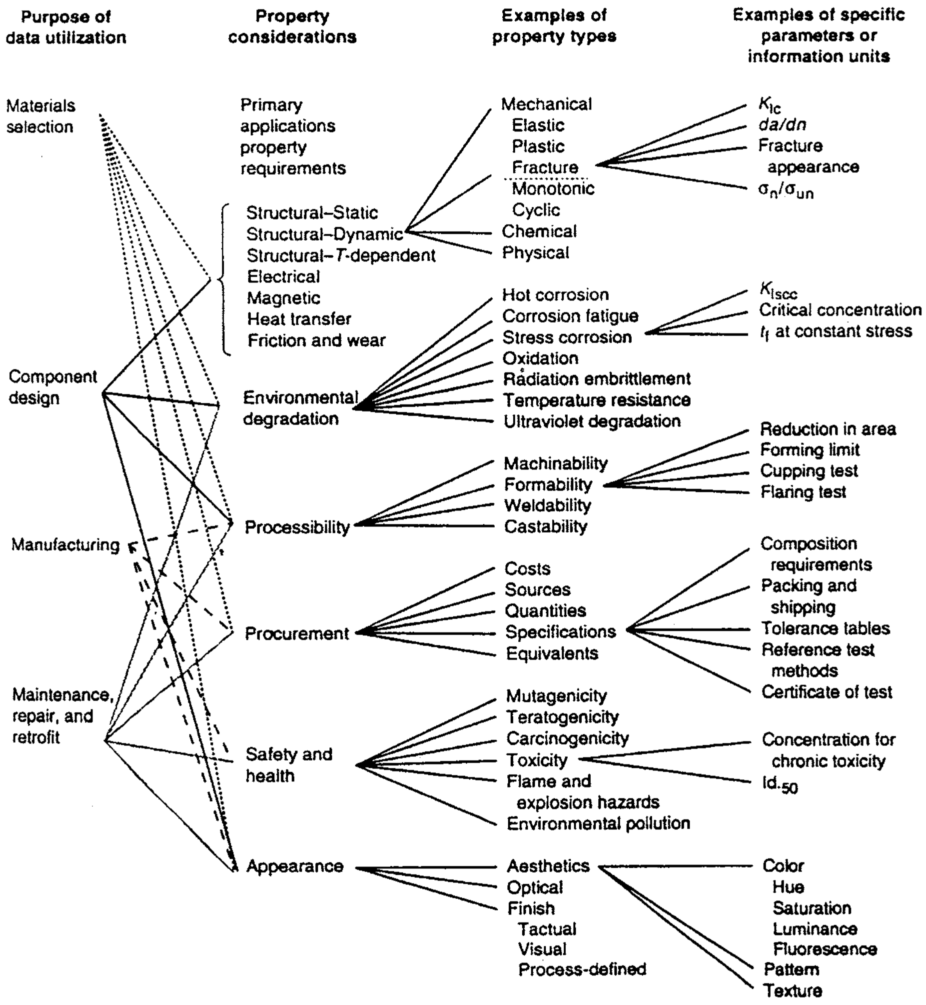

Fig. 1 Taxanomy of materials information. From *ASM Handbook, Vol. 20: Materials Selection and Design (1997),* ASM International, Materials Park, OH 44073-0002, fig. 1, page 491.

factors that go into judgments of data quality and reliability.[10] There are two parts to such a discussion: (1) the two major factors affecting the data themselves and (2) the degree to which those factors are reflected in database content.

The two factors affecting quality and reliability include (a) the manner in which the data were obtained and (b) the statistical reliability of the data presented.

First and foremost, the users of a database for whatever purpose will want to know that consistent standards were applied in assembling the data for that database, and that the properties of one material may reliably be compared with those for another. They would also prefer that those properties have been generated by well-known standard methods such as those prescribed by organizations such as ASTM and ISO.[4] They may also be quite interested in knowing the specific source of the original data; realistically some laboratories [e.g., the National Institute of Standards and Technology (NIST)] have a more widely recognized reputation than others.

The second major factor affecting how the user applies information from a specific database is the statistical reliability of the values included therein. The user will be interested in which of the following categories best describes the values presented:

- Raw data (the results of individual tests)
- Average of multiple tests (how many tests represented?)
- Statistically analyzed (at what statistical definition and with what confidence?)
- Evaluated/certified (by whom? for what purpose?)

Of equal importance to the user of any database for whatever reason is the degree to which those factors affecting quality and reliability as noted above are presented in the database itself and, therefore, are fully understood by the user. In some instances there may be one or several screens of background information laying out the general guidelines upon which the database was generated. This is particularly effective if the entire database represents properties of a common lineage and character. On the other hand, if the user fails to consult this upfront information, some important delimiters may be missed and the data misinterpreted.

Another means of presenting the metadata concerning quality and reliability is within the database itself. This is especially true for such factors as time–temperature parameters that delimit the applicability of the data and units and other elements of information that may restrict its application (see Section 3.3).

It is also especially important in instances where the individual values may vary with respect to their statistical reliability. While the later case may seem unlikely, it is actually quite common, as when “design” data are presented: in such cases, the strength values are likely to be statistical minimum values while moduli of elasticity and physical properties are likely to be average values, and the difference should be made clear in the database.

6 PLATFORMS: TYPES OF DATA SOURCES

The last feature we will consider before identifying specific data sources is the variety of platforms available for databases today. While it is not necessary to discuss them in detail, it will be obvious that the following choices exist:

- Hardcopy (published books, monographs, etc.)
- Self-contained electronic (floppies, CDs, etc.)
- Internet sites (available online; perhaps downloadable)

The only amplification needed on these obvious options is that the last one, the Internet, has become an interesting resource from which to identify and locate specific sources of materials data, and that trend will likely continue to increase. Two large caveats go with the use of Internet sources however: (1) a great deal of "junk" (i.e., unreliable, undocumented data) may be found on Internet web sites, and (2) even those containing more reliable data seldom meet all users needs with respect to covering the metadata. It is vital that the users themselves apply the guidelines listed earlier to judge the quality and reliability of sources located on the Internet.

7 SPECIFIC DATA SOURCES

It is beyond the scope of this chapter to provide an exhaustive list of data sources because there are thousands of them of varying presentation platforms, styles, and content. What we will do here is highlight a few of the potentially most useful sources, in the sense that their coverage is relatively broad and/or they represent good places to go to look for new and emerging sources of materials data.

The categories discussed below will include:

- ASM International, a technical society for materials engineers and specialists that produce and provide a wide range of materials databases in hardcopy and electronic form (see Section 7.1)
- STN International, a service of the American Chemical Society, also providing (for fee) access to a very wide range of numeric databases as well as a great many textual/bibliographic databases on materials-related subjects (see Section 7.2).
- The Internet, the most rapidly expanding source for materials data in a variety of forms (see Section 7.3).

For readers interested in more extensive listings, reference is made to the article "Sources of Materials Property Data and Information" by Jack Westbrook in Volume 20 of the *ASM Handbook*[1] and to the *ASM Directory of Databases*.[3]

7.1 ASM International

ASM International has emerged as one of the strongest providers of numeric materials data sources, and those sources are generally in at least two formats or platforms: hardcopy and disk, usually CD-ROM. As an example, one of the most extensive sources of high- and low-temperature data for aluminum alloys has recently been made available through a collaborative effort of ASM and the Aluminum Association, Inc., in both the book *Properties of Aluminum Alloys—Tensile, Creep, and Fatigue Data at High and Low Temperatures*[11] and a searchable CD of the same title. Other representative data sources from ASM International include:

- *ASM Handbook,*[12] in hardcopy and CD—20 volumes complete or available in a specific set covering material properties; the data sets are available for single workstations and also for local-area network (LAN) arrangements. Other CDs are available covering heat treatment, testing and analysis, and manufacturing processes.
- Alloy Finder CD—contains full alloy records from three ASM hardcopy reference standards: *Woldman's Engineering Materials,*[13] the ASM *World-*

wide Guide to Equivalent Irons and Steels,[14] and the ASM *Worldwide Guide to Equivalent Nonferrous Metals and Alloys.*[15] The disk is searchable by composition as well as designation, so whether the user requires amplification of an alloy designation or to locate designations within specific composition ranges, the need is addressed.

- *Alloy Digest* on CD—Summaries of recently published data for new and emerging alloys are compiled periodically on disk as well as being made available in hardcopy. The advantages include early warning of new materials; the limitation is the inability to provide consistent formats or data scope because such are not available for relatively new materials. More than 4200 data sheets are now available.
- Binary Phase Diagrams on CD-ROM—The world's most extensive collection of binary phase diagrams numbering in excess of 4700 is available on CD, in addition to ASM's hardcopy publications such as *Handbook of Ternary Alloy Phase Diagrams* and the monograph series on specific alloy groupings.
- Failure Analysis on CD-ROM—One of the most extensive collections of data expressly developed for failure analysis is available on searchable CD-ROM as well as hardcopy.
- Materials databases on various classes of materials are available from ASM on disk in various formats including Mat.DB, MAPP, and Rover search software. The databases are organized and searchable by mechanical and physical property as well as alloy class: steel and stainless steel, aluminum, composites, copper, magnesium, plastics and polymers nylon, and titanium, plus a special disk covering corrosion data.

For more detailed information on ASM International's data sources, the reader is referred to its Internet web site: www.asminternational.org.

7.2 STN International

STN International is the online worldwide scientific and technical information network, and one of the most extensive sources of numeric materials property data.[16] Built and operated by the American Chemical Society at its Chemical Abstract Service site in Columbus, Ohio, STN International includes about 30 numeric databases, including many developed during the collaboration with the Materials Property Data Network.[17–19] Of great interest to materials data searchers, STN International has the most sophisticated numeric data search software available anywhere online. Thus the data sources may be searched numerically, i.e., using numeric values as ranges or with "greater or less than" types of operators, making it possible to search for alloys that meet required performance needs.

The disadvantages of using STN International to search for numeric data are twofold: (a) the search software is keyed to a command system best known to professional searchers, and engineers and scientists will need patience and some training to master the technique; and (b) use of the STN International system is billed via a time and content based cost accounting system, and so a private account is needed. Outright purchase of databases has not been a policy.

For those able to deal with those conditions, a number of valuable databases exists on STN International, including the following representative sources:

- ALFRAC—Aluminum Fracture Toughness Database (1968–1989 industry compilation)
- Aluminium—Aluminum Industry Abstracts (textual; 1868–present)
- ASMDATA—ASM Materials Databases online version
- BEILSTEIN—Beilstein Organic Compound Files (1779–1999)
- COPPERDATA—Copper and Copper Alloy Standards & Data
- DETHERM—Thermophysical Properties Database (1819–present)
- DIPPR—AIChE Design Institute Physical Property Data File (1982–present)
- GMELIN—GMELIN Handbook of Inorganic Chemistry (1817–present)
- ICSD—Inorganic Crystal Structure Database (1912–present)
- NUMERIGUIDE—Property Hierarchy and Directory for Numeric Files
- MDF—Metals Datafile (1982–1993)
- PIRA—PIRA and PAPERBASE Database (1975–present)
- PLASPEC—Plastics Material Selection Database
- RAPRA—Rubber, Plastics, Adhesives, and Polymer Composites (1972–present)

For more information on STN International, readers are referred to the website www.cas.org.

7.3 Internet

As indicated earlier, there are many web sites on the Internet with materials information content. The challenge is to determine which have useful, reliable, and relatively easily accessible data. In the interests of readers, we will focus on guidance on these latter points developed by Fran Cverna, Director of Electronic and Reference Data Sources at ASM International, who recently produced and presented a survey of the scope and quality of materials data content found on Internet web sites.[20]

Among the most useful web sites of the several hundred screened by Cverna and her ASM resources are the following:

www.about.com	Internet search engine—search Materials and Properties
www.nist.gov/public affairs/database.htm	NIST—standard reference database
www.ecn.purdue.edu/MPHO	TPRL—thermophysical properties data
id.inel.gov/shds	U.S. government—solvent database
www.campusplastics.com	Campus consortium—plastics database

www.copper.org	Copper Development Association—copper alloys database
www.brushwellman.com/homepage.htm	Brush Wellman—supplier materials database
www.timet.com/overviewframe.html	Timet—supplier materials database
www.specialmetals.com	Special Metals—supplier materials database
www.cartech.com	Cartech—supplier materials database (compositions)
www.macsteel.com/mdb	McSteel supplier materials database (limited)
www.aluminum.org	Aluminum Association applications—publications' limited data

Of the sources above, two merit special mention, those being the Internet search engine site www.about.com and the National Institute for Science and Technology [(NIST) databases site, a part of the public affairs menu].

The about.com site provides an excellent means of locating materials data sites and provides a subcategory called "Materials Properties and Data" if you search for such information. Thirty-two sites are identified, some of which overlap the ASM survey, but others are unique to that site in my experience. Many included in the ASM survey are not included here, so the two are supplementary in scope. Among the specific types of information accessible via about.com are:

- Coefficient of thermal expansion
- Electrical conductivity
- Electrical resistivity
- Hardness conversion charts
- Metal temperature by color
- Metal weight calculator
- Periodic table of elements
- Specific gravity of metals
- Surface roughness comparison charts
- Thermal properties
- Wire gauge conversion tables
- Utilities to simplify tasks such as conversion charts for units, currency conversion, glossaries, acronym definitions, etc.

In addition there are links from about.com to many other materials sites, including one called aluminum.com that also provides materials data for a variety of metals, but often without adequate citation and metadata.

The NIST database site provides direct online access to the highest quality, carefully evaluated numeric data from the following databases:

- Standard Reference Data—reliable scientific and technical data extracted from the world's literature, assessed for reliability and critically evaluated
- Ceramics WebBook—evaluated data and access to data centers as well as tools and resources
- Chemistry WebBook—chemical and physical property data for specific compounds
- Fundamental Physical Constants—internationally recommended values of a wide range of often used physical constants
- Thermophysical Properties of Gases for the Semiconductor Industry

To summarize the Internet discussion, there are many sources of numeric materials data available from Internet sites. It remains for the potential users of the data, however, to approach each site with caution, look for the pedigree of the data in terms of quality and reliability, and to make certain that the source used meets the requirement of the intended use.

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CHAPTER 17

MANAGING MATERIALS DATA

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Previous chapters have described the sources and usage of materials data in product manufacturing organizations. This chapter discusses the process of managing materials data. After examining its history, discussions will be provided on the design and implementation of materials data management systems, the design and creation of the underlying database(s), and the evolution and use of standards for materials data reporting and exchange. A brief summary of commercially available database management systems is provided. We will begin by establishing the premise that the ability to manage the vast amount of materials information used in manufacturing organizations is perceived as a tremendous tool for increasing efficiency and profitability.

Materials data is used to support many different functions in a manufacturing organization from the initial phases of design to the disposal requirements at the end of a products' life cycle. The most critical of these functions is the process of analysis, used to predict the product's performance in its end-use environment. The completeness of the required data sets is crucial to most forms of mathematical analysis required for engineering simulation. The pedigree or background data for the tested material property data, as well as the specific properties required for the analysis must be readily available to the analyst. These properties are either acquired from established sources or the result of rigorous testing or a combination of the two. It has been estimated that the cost

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Handbook of Materials Selection. Edited by Myer Kutz

of testing for a complete set of materials properties required by a designer for a new application can be in excess of $1 million with an acquisition time of greater than 5 years.[1] Additionally, it is estimated that engineers spend over half their time acquiring and verifying the validity of this data. Effective management of this data is paramount for organizations having functions that are materials data intensive. It reduces the cost of materials data acquisition, improves the usability and reusability of acquired data, and increases the efficiency of the engineering organization.

Historically, the confidence in a new product design relied on large safety margins and field service experience. The introduction of computerized design and analysis systems offered the promise of selecting the right material for an optimized design early in the product development cycle. This requires that consistent and high-quality materials information be used throughout the design to manufacturing process. In addition to the resulting cost reductions, the benefits of effective data management are realized in terms of higher quality, decreased warranty costs, shorter product development cycles, and traceable and auditable systems.[2]

So what does "materials data management" mean to an organization? It means:

- Making the materials properties acquired by an organization available to all functions requiring that data within the organization in a consistent and timely manner from an official, centralized source
- Providing a means of controlling the sources of materials properties used for a given function
- Controlling access to specialized and proprietary information
- Providing the available materials properties in a format that optimizes its usability and reusability

1 HISTORY OF MATERIALS DATA MANAGEMENT

In many organizations, the engineer responsible for a specific analysis maintains the information he requires. It is often found in old notebooks, jotted on scratch pads, in textbooks, and other references known only to him. This data provides little, if any, traceability and limited pedigree information. The values are often just simply those that have "always been used." If the engineer leaves the organization, the search for the required data begins again. Often the data is not accessible to other individuals within the same company, and the acquisition process is duplicated, sometimes resulting in the use of different values for identical properties, producing inconsistencies in analysis of the same system or structure. It has been estimated that 50% of an engineer's time is spent searching for and verifying materials property data.

Organizations for whom traceability is important often require "certified" data, which has been evaluated by experts and approved for a specific application. These organizations have long recognized the value of the materials data they use. Many organize the data into official handbooks that are distributed throughout the organization, fulfilling the requirement for controlling the data used in their analysis processes. However, the process of creating the handbooks is time-consuming and costly. Rocketdyne Division of Boeing North America documented the process for adding data from a vendor for a new material into its handbook[3] in the following manner:

1. Test data obtained from vendor was typed into a curve-fitting program.
2. A best-fit curve was constructed based on the test data.
3. Archived data and data from other sources for the same material and test condition were retrieved to compare with the newly constructed curve.
4. If a difference existed between the new data and historical data for similar materials, the difference was reconciled in one way or another, and the new data was finalized.
5. A written report was generated for distribution to those who immediately needed the data.
6. The report was archived in the files.
7. The newly constructed curve would eventually be entered in the handbook.

Since the hardcopy manual was difficult to update, this process may have taken up to a year. Rocketdyne's hardcopy materials properties manual consisted of four volumes with over 1500 pages and 5000 curves. Revisions to this manual involved a major effort in terms of time, as well as paper.

As usage of computers became more widely available, companies began to realize the potential for tremendous cost savings through the optimization of their processes. They envisioned the capability for systems that would allow them to go beyond mere data storage within their organizations. The specialized use of computers in storing materials data for access by designers and analysts companywide provided new capabilities to search for and compare materials based upon specified properties and allowed designers to consider new materials options early in the design phase of a new product cycle. The possibilities for shortening the design cycle, making products of higher quality, improving the consistency of manufactured parts became a reality. Most of these improvements were driven by the need to improve profitability, others by the need to provide audit trails as required for ISO 9000 compliance. Response to these new requirements has led to the development of numerous "materials databases" in the past decade, either as commercially available products or in-house developments.

The introduction of product data management (PDM) systems to the marketplace provided additional tools for the integration of product data throughout an organization. While slow to catch on, by 1999 the estimated market size for PDM systems reached $1.6 billion. Its growth was driven by an increasing awareness of the value of these systems in improving workflow, configuration management, document and information integrity, and communication throughout the enterprise.[4] Various specialized functions, including materials data management systems, are now being linked with PDM systems. For the manufacturing community, PDM is a natural complement to computer-aided engineering (CAE), by providing a repository for data that is accessed by CAE. The integration of CAE and PDM enables the access, verification, and control for

CAE input and output data.[5] "Simulation data management" is vital to the integration of CAE into the product development process. The benefits are analogous to those of materials data management systems and include:

- Improved access to authorized data sources
- Higher throughput for analysts and designers, resulting in faster and more reliable engineering decisions
- Fewer simulation errors, increased repeatability of simulation methods, with full traceability
- Enhanced collaboration between design and simulation, within the enterprise and throughout the supply chain

The Internet, or World Wide Web, has been credited with the substantially increased market for PDM-type systems. Being universal, inexpensive, accessible, and hardware-independent, web-enabled PDM formed a prototype for web-enabled materials data management systems. Even in organizations where the databases are not fully integrated, and legacy systems remain, web enablement allows the data from multiple sources to be displayed on the screen at the same time. This includes not only data from the PDM system but from the shop floor, parts management systems, purchasing, finance, shipping, and legacy systems as well. Once accessed, a web interface, with the look and feel of standard reporting mechanisms, can be used to generate reports by simply printing the screen. Most companies find that establishing a workable web browser interface as a front end to their PDM system is quite simple, requiring minimal programming expertise and technical support.[6]

While the Internet provides access from or to vastly different sources, the Intranet has enabled organizations to distribute materials information from a centralized location to all its satellite offices and divisions. This bourgeoning expertise has facilitated the distributed "build-design" teams in many global enterprises that support the "design anywhere, build anywhere" business model.

As use and acceptance of these systems expand, additional applications have surfaced in the area of business-to-business collaboration. The power of the Internet allows companies to readily transact with each other and access each other's information, making collaboration a reality. The first wave of Internet collaboration was in the supply chain, as companies worked with customers, suppliers, and intermediaries to manage inventories. By simplifying transactions and sharing information, companies continue to drive down costs, cut cycle times, and reduce inventories. The cutting edge of supply-chain collaboration is moving upstream from there. Intermediaries and suppliers are now doing collaborative planning and forecasting, creating a shared vision of what customers will eventually buy, so that the right product can be in the right place at the right time. Companies are also collaborating on product development. Instead of a system maker and a component supplier developing their products separately and then trying to make them fit, they use a shared design database to develop everything together from the outset. In some industries, competitors are standardizing on common parts and practices by sharing information over the Internet. Companies must be prepared to work with others—not out of a sense of altruism, but for shared benefit.[7] Today, an array of "off-the-shelf" systems and custom implementations are available, making collaborative PDM tools convenient and economical for even small and medium-size companies.

2 IMPLEMENTING A MATERIALS DATA MANAGEMENT SYSTEM

Based on the previous discussion, it is apparent that the primary drivers for materials data management in today's industries are the requirements to:

- Reduce the time to manufacture and the associated need for quick access to approved data.
- Disseminate or exchange data between different functions or organizations.
- Increase product quality and consistency resulting in lower warranty costs.
- Increase profitability by decreasing design, manufacturing, and testing costs.
- Improve traceability of data pedigree to substantiate product warranty claims.

Therefore, a materials data management system must be more than just a computerized version of a set of handbooks.[8] It must serve the material information requirements of the entire product life cycle. These include:

- *Conceptual design,* where materials information is required to screen candidate materials based on functional performance requirements and prior experience with candidate materials used in similar products.
- *Design,* where materials information is needed for specific (stock) materials, their grades and finishes, standard components and/or joining materials as specified in the engineering bill of materials. At this stage, specific material objects are designed and analyzed for end-use environments including manufacturing, assembly, service, maintenance, and disposal.
- *Manufacturing,* where materials information is needed for specific material object manufacturing processes, tooling, assembly fixtures, joining materials, dunnage, and in-process materials consumed during manufacture.
- *Operation,* where materials information is needed for in-service operation, including data for health monitoring, maintenance, repair, and other material-specific operating information provided by the manufacturer to the end-user.
- *Disposal,* where materials information on composition, procedures for recycling, and other environmental impact data are required at the end of the product's service life.[9]

The success of a materials data management system lies in appreciating that the end users at each step of the product life cycle must access, retrieve, and manipulate approved materials data, and that the system structure, its user interface, and its data content must be designed to provide for this wide range of usage at the outset. A well-designed and integrated materials data management system should provide efficient archiving and distribution of knowledge, eliminating duplicate materials testing, and optimizing the number of specified materials.[10]

Most current configurations of materials data management systems use client-server technology, incorporating a centralized database management system (DBMS), software applications that enhance the DBMS, and web access. As discussed earlier, the flow of data throughout the organization, exported in a usable format to each function may be enhanced by PDM configurations that interact with the materials data management system. Then the materials data becomes integrated throughout the organization. Otherwise, web enablement can provide easy access to the materials data management system by simple entry of a URL in a web browser window. The web-enabled materials data management system is the focus of the following discussions.

2.1 Planning

A successful implementation requires full management commitment and careful planning, which includes the following elements:

- Clearly identifying the vision for a successful implementation, stated in terms of the company's business objectives. The vision statement is used to guide decisions for purchases and implementation and to define the metrics for success.
- Creating a project plan, resource allocation plan, and time allocation plan.
- Risk analysis and mitigation throughout all phases of the implementation.
- Planning and managing deployment and closure of each project and subproject.

2.2 Implementation

Implementation of a materials data management system is a customization of standard software development practices. The four phases in implementing a new software solution include analysis, design, implementation, and testing. The following sections provide information regarding the application of this process to the design of a materials data management system within a manufacturing organization. Implementation and testing, therefore, is followed by deployment of the software throughout the organization and system support.

Analysis

The initial discovery phase provides the backbone for the entire implementation. Its goal is to understand the organizations' compelling needs and/or primary business drivers for implementation of materials data management, how it should function within the organization, and how the system should be deployed. Statements similar to the following represent typical business drivers:

- To reduce cost and process cycle time by a factor of 10
- To improve communication, integration, and flexibility

- To promote innovation and understanding of the use of new materials
- To improve productivity by running all operations from a single materials database
- To eliminate redundant materials testing by making data available to all analysis functions

This initial phase must first define and document the company's product development process and how materials data is currently used in that process. The use of detailed process charts for the mechanical design and analysis of the product can clearly identify the data flow requirements and allow for standardized methodologies.[11] All end users of the system and/or end-use applications with which the system must be integrated should be identified during this process. This may include the following materials-related functions:

- Product design
- Engineering analysis
- Materials engineering
- Manufacturing specification
- Materials ordering or bill of materials
- Other applications, such as PDM or collaborative PDM systems
- Functions at satellite offices

For each function, the following should be identified:

- The current data sources
- The types of data to be stored in the database
- How the data is retrieved and viewed
- The form the data must be in to be read by the individual applications
- How data it is to be reported or extracted from an application
- What standards and terminology must be utilized by the system
- Security or controlled access requirements
- Versioning control requirements

This preliminary requirements gathering provides an understanding of what schemas, databases, user interface requirements, queries, and other customizations may be required within the end user's environment. It also provides an indication of the internal and external resource requirements. The project may be further promoted by assessing, near term and long term, the cost of inaction.

Deliverables: Preliminary findings document, vision statement, and the data flow diagram.

Design

The system design phase must produce a detailed set of requirements for the system. Software requirements include the definitions of the user interface software, the DBMS, and any software required for versioning or controlled access to the data. Hardware requirements should include preferred platforms for both client and server. From this, an initial materials information solution can be proposed and a return on investment (ROI) analysis provided.

Selecting a DBMS requires understanding your organizational requirements. While databases are traditionally designed to simplify the development of data-intensive applications, a carefully selected database management system can eliminate development altogether, by providing enough functionality that the end user can use the tools immediately to enter, examine, and process application data.[12] Traditional database requirements include:

- The ability to store a large volume of structured data
- Capability to retrieve data from storage via a simple query mechanism
- Distributed capability, allowing data storage on more than one location or machine
- An applications package, providing easy data modeling capability
- A crash recovery system, providing the ability to bring the database back to a consistent state automatically
- Data independence, allowing data changes without modifying existing application programs
- Reasonable performance requirements

Additionally, the DBMS used to store materials property data must have the capability to store and handle complex data types, including numerical values with units, footnotes, and numerical precision, graphical data, images, and documents.

The user interface software for a web-enabled materials data management system must typically provide the following capabilities, most of which will be identified in the discovery phase:

- Graphical displays for comparison of materials properties
- Search capabilities based on product names (character strings) or properties (numerical values)
- Interaction with data pre- and postprocessing applications, such as Excel or other proprietary software
- Ability to add to or delete data from the database
- Ability to extract or translate required data sets to identified applications, in required formats
- Ability to utilize existing standards for data modeling
- Ability to convert units between standardized unit systems, ensuring consistency in an analysis and compatibility with analysis or postprocessing software
- Customizability of the user interface, using common web interface methods and technology such as HTML and/or Java programming.[13]
- Versioning control or provision for a formal update process to ensure that a user can verify use of the most current data

- Secured or controlled access to subsets of stored data. Compilations of engineering knowledge, such as proprietary materials property data, represent a substantial investment for manufacturing companies, access to which must be carefully controlled. This is particularly challenging in global markets, where manufacturers may be partners on one project and competitors in another.[13] When dealing with the Internet or Intranet and PDM, both PDM security access and transmission security must be implemented. Outside a company's firewalls, transmissions can be secured by encrypting files in the vault prior to transmission. Within companies, usage can be secured using standard secured access processes.[4]
- Integration with other systems via PDM, application programmatic interface (API) or integrated client technology.

Deliverable: Preliminary statement of requirements.

Implementation

The implementation process is generally scheduled over a period of time, phasing in the database building process with implementation by the various functions requiring access to the data. This process can be facilitated logically by the use of the data flow diagram constructed in the initial design phase. It is often helpful to identify low-risk, high-return pilot implementation opportunities. The tasks for this phase include:

- Definition of implementation deliverables and milestones
- Identification of process redesign necessary prior to deployment
- Development of a timeline to achieve a total implementation (the implementation plan), including both pilot and fan-out
- Need for documentation and training
- Development of an executive summary based on results for reference by management
- Estimates of the return on investment (ROI), based on the implementation plan

Deliverables: ROI Projections, requirements document (user and high-level technical requirements).

Testing

This phase includes the installation of hardware and software for a specified portion of the organization or a specific function. To provide for a thorough evaluation of the software, a prototype database must be designed, built, and installed. The user interface must be designed or customized, and training materials created. Extensive testing must be performed on this initial concept of the solution. Usage, performance, support requirements, user issues, and data issues should be documented. From this, modifications to the software will be identified and implemented. Resource requirements can be refined, and the details for fan-out can be specified and adjustments made as required. ROI projections can be revised accordingly.

Success at this stage can be measured by the ability of a team to quickly integrate the system into their day-to-day activities, documented time savings in retrieving data, exporting data to specified applications, comparing materials, and meeting reporting requirements.

Deliverables: Design document, and a functional application to be scaled into a production application in the next phase.

2.3 Deployment and Support

At this phase, upgraded software is installed at the pilot installation site, verified, and evaluated. Customization of the user interface is refined. Training should be extended to include the remaining user and administrative community. Database production procedures should be developed and implemented. Database building is expected to continue, but the schema design should be finalized based on user experience.

If the upgraded implementation is satisfactory, fan-out is continued based on the adjusted implementation plans. In the case of web-enabled access, the application software can be installed on the designated servers and accessed from client machines. The web browser interface should be refined based up the most up-to-date customized version of the software application. The requirements for ongoing support should be defined.

Success is measured by centralized access to approved materials data for all specified engineering and manufacturing functions. The majority of end users should feel comfortable with the revised materials processes. Materials-related activities should exhibit increased efficiency.

Deliverables: Customer training/administration/user manual(s) and production application with complete production data integration.

3 CREATING THE DATABASE

The following is an overview of the process used to design and create a materials database that best meets the specifications of the end users. It is not the intent of this section to provide an exhaustive study of database design and implementation but only a general understanding of the scope of the tasks for creating a database useful to a manufacturing organization. There are many excellent resources available on this topic, from extremely technical to hands-on resource guides. In most instances, the reference guides provided with DBMS software adequately describe the technical details of database design required to physically create a database for use with that application.

The success of any project, especially that of creating a materials database from numerous sources of data, requires careful planning and the commitment and organization provided by a project team. The approach outlined in this section requires defining the database in the context of the individual end-use applications to assure that all end-users have as complete a data set as possible. The requirements for each end-use application may then be merged to create one database. After the database is compiled, the interface can be customized to optimize use of the database by the various end users.

3.1 Defining the Project Team

In creating the project team for designing and creating the databases, the following functions are suggested. It is highly recommended to have one individual, generally the project leader, who provides the focal point for all communications involved in the database design and building process.

The *project leader* must champion the cause, organize the project, and coordinate the effort. This person sets the milestones, establishes acceptance criteria, periodically reviews the project, and puts in place the approval procedures.

The *end user* defines the end use of the database, which must supply all necessary information in a usable form. Therefore, the end users should be involved in every level of database design and development. Their input determines what materials and what information for each material are stored in the database, how the data is stored, how the data is retrieved, how the user interface is customized, how the data is exported, and what unit conversions are required. Note that a database may be developed primarily for the organization and storage of data. In this case, the end user is often also the data provider.

The *data provider* is a focal point for all data sources. This person is responsible for gathering the information from all data sources required to meet the needs of the end users. He compiles the data, identifies and documents the storage media and format, and organizes it for entry into the database. Input from the data provider helps to determine the most efficient methods for data entry.

The *programmer* is responsible for translating electronically formatted data into formatted files for loading data into the database. This person is also valued for his expertise if data reduction or manipulation is required prior to data entry.

The *database builder* creates the database using the tools provided by the DBMS application. The database builder compiles all the information provided by the end users, creates the schema design, creates or compiles the data loading files and/or techniques, initializes and loads the database, creates all required auxiliary files, and corrects errors discovered during the quality assurance process.

3.2 Defining the End User Data Requirements

While efficient data storage may seem to be the purpose of a database system, the utility of the database is severely limited if it is not designed to meet the needs of the end use application(s). Therefore, a clear understanding of both the short-term and long-term end user applications is required.

For each application identified in the initial planning phase, specify the following materials data storage requirements should be specified:

- What materials are to be included?
- What information is applied globally to all end users?
- What pedigree information is required for each material?
- What properties are required for each material to optimize use of the database by the end user?
- What information is best provided as footnotes or metadata? (i.e., it is too sparse to require an attribute or too irregular to categorize)

- What are the user access privileges? Is some data restricted?
- What assumptions must be avoided?
- How often must this data be updated?

3.3 Defining the Functional Requirements

Most of the functional requirements are a result of or defined by the end user application. When defining the functional requirements, try to anticipate future applications for the database. Typically, the following questions should be answered:

- What types of queries (groupings of subsets of data) or data searches, list them if possible, will be performed on the database?
- What information is required to adequately identify the material records returned by the query or search?
- Will curve, table, or matrix data need to be represented?
- Will data be mathematically manipulated, required by pre- or postprocessing, and what is the required accuracy?
- What information is routinely accessed?
- Will the data be exchanged with or exported to other applications?
- What will be the acceptance criteria? Define the individuals responsible for approval.

3.4 Designing the Database

Materials data management systems typically have similar interface requirements, but the underlying database will be one of three common structures: relational, hierarchical, or a hybrid of the two. The following discussion relates to the process of creating a hybrid hierarchical-relational database. Comments are provided for their adaptation to relational databases.

Before beginning the database design, it must be determined whether to store the data in one database to be accessed by all end users or to divide the data into separate databases. This relates to decisions regarding the requirement for a distributed database system. Note that data can be stored in one database and then subdivided into separated databases for the individual end use applications. This often makes the process of updating the databases simpler. Some of the questions to be answered include:

- Will the total volume of data be unwieldy? Smaller databases are always faster and easier to update.
- Are the end uses similar or radically different? Is one or many databases expected by the end user?
- Will the data sources be updated on disparate schedules (i.e., archival data vs. ongoing test data)?
- How well do the categories of attributes that define the materials from different end users overlap? Can multiple databases use the same schema?

- Are the materials properties tested to similar standards?
- Does the database size pose a problem for any of the end users based on performance requirements?

Having developed an understanding of the end users and how they intend to use the database and data, it can then be determined which elements are to be stored in the database (or each database). More data may be available than is required to meet the end user requirements. Data that is not specifically required by any of the end users should be evaluated for its potential utility before being added to the database. Often it is desirable to keep all available data in one place as an official repository.

Once all data elements are listed, the relationship between the elements is established. This information is used for the development of the database schema, which defines how the data is structured within the database. The following information, preferably compiled in sequence, is required for *each application.*

1. *The Data Elements* (*Attributes*). Database attributes are a numerical, textual, or graphical representation of the data. Textual attributes are useful for storing documents related to materials, tests, test data, property data, engineering specifications, revision histories, etc. Images are often included to demonstrate test layouts, engineering drawings, photomicrographs of test results, photos of specimens or products, etc.

All attributes that are required to provide the end user with the information necessary to qualify material property values and to provide the property values themselves for the materials in the database must be identified. These attributes should serve one of the following functions:

- To characterize the material to the level required by the user. The American Society for Testing and Materials (ASTM) and other organizations provide standards for accurately characterizing materials.
- To identify the source of the data. Data provided by different organizations may include the name of the organization, address, phone, test employee identifiers, etc. Validated data may require different parameters than test qualification. In-house or in-process test data may include the test group and employee identification or similar parameters for tracking purposes.
- To identify test parameters, conditions, methods, and the details of the test specimen. ASTM provides standards for identifying and qualifying test methods.
- To identify the property values to be stored in the database for the materials. This includes single-point scalar and test values, curves (arrays), images, and documents.

2. *The Attribute Definitions.* The attribute names and definitions provide a dictionary relating the attributes to terminology easily understood by the end user. These names and definitions are used in the schema that defines the database structure, to create customization files, and in quality control procedures.

Relational databases typically use field specifications to define attributes. Field specifications are composed of five types of elements: general elements, physical elements, logical elements, and specification information. Field-level and data-level integrity are established using thorough definition of the field specifications for each attribute.

Attribute definitions should include, as a minimum, the following information for a materials database:

- *Attribute Type* (Physical Element). The attribute type determines the form by which data can be searched and how it can be manipulated. This may include character strings, integers, real numbers, curves, matrices, text files, images, etc.
- *Units* (Logical Element). A consistent unit system must be provided for the entire database. The units must be consistent for each instance of an attribute in the database. Unit conversions may be required to provide the end user with useful information in the units system required by his application.
- *Description* (General Element). The attribute should provide a description of the attribute name in terms the end user understands. This label is typically used in displaying and searching data.
- *Numerical Precision* (Physical Element). The attribute precision provides the number of significant digits to which data is to be stored in the database and number of significant digits to which data will be displayed. This applies to all real, curve, and matrix values. American Iron and Steel Institute (AISI) provides standards for precision of computed values.
- *Constraints for Allowable Values* (Logical Element). Determine and record data constraints to identify out-of-range data during the data entry process. Also determine existence constraints when the data must be supplied for a material or property in order for it to be a valid entry in the database. This information can be used for data translation, database building, and quality assurance.
- *Attributes for Thesaurus or On-Line Help* (General Element). Identify information relating to the attributes that should be provided as end user documentation.
- *Test Specifications* (General Element). Identify and record test specifications, conditions, or identification that applies to all instances of property attributes.
- *Quality Indicators* (Specification Information). Quality (statistical) indicators or qualifiers required to qualify the data for the end user should be provided for property data. For example, the National Institute of Standards and Technology (NIST) provides the following guidelines for use of its on-line Ceramics WebBook, a collection of property values derived from surveys of published data[14]:

  Certified (standard reference values)
  Validated (confirmed via correlations and models)
  Evaluated (basic acceptance criteria satisfied)
  Commercial (manufacturer's data)

Typical (derived from surveys from values for nominally similar materials)

Research (preliminary values; work in progress)

Unevaluated (all other data)

3. *The Attribute Groups.* Having identified the attributes to be included in the database, they are now grouped by common characteristics in the context of the end user. Typical groups of attributes are specimen identification, material composition, specimen condition, specimen preparation, test conditions, mechanical properties, electrical properties, physical properties, etc. These groups of attributes are referred to as relations or tables.

It is important to verify that all the attributes belong to an appropriate relation. If the database has more than one end use application, the requirements for each are integrated, while avoiding redundancy. Additional tables may be added to improve data normalization.

Purely relational databases require the addition of key attributes to each relation that uniquely define each row in the table. These keys are used to link the tables for the purposes of uniquely identifying a material in the database and querying information from the database. Hierarchical databases use a combined set of attribute values for a material stored in the database to uniquely define the material record.

4. *The Relation Names for Attribute Groups.* A name must be assigned to each group of attributes using a term that best characterizes the grouping. Relation names are very important in relational databases, as they are specified in the SQL queries used to retrieve data from the database. Therefore, they must be unique, descriptive, and unambiguous. Relation names are not as important in hierarchical databases because they are not typically used in querying, but it is good practice to maintain their uniqueness.

5. *The Relationship between Relations.* For databases with a hierarchical structure, the relationship between the groups of attributes defines the hierarchy (parent–child relationships) of the database, dictating the structure for the schema design. This hierarchy provides the path to the property data. The relations should be separated into two groups: those that identify characteristics for the materials and those that specify properties of the materials. The tables storing defining characteristics are typically ordered according to the level of material differentiation the data provides, from the least differentiation (i.e., material, test type, component) to the highest (i.e., test conditions, source data). The relations that contain attributes that define the actual property data are grouped into tables by type and are typically on the same level at the bottom of the hierarchy.

For relational databases, the relationship between the groups of attributes establishes the connection between relations that are logically related to each other in some form or manner.[15] These relations are used to refine table structures, minimize redundant data, allow data to be drawn together simultaneously, and provide for relationship-level integrity.

Hierarchical database relations generally have one-to-many relationships progressing down the levels of the schema. Relational databases usually have one or more of three types of relationships: one-to-one, one-to-many, and many-to-many.

6. *The Schema.* The information gathered in the previous steps is organized to create the schema for a hierarchical database or can be used to establish the relationships and create the field specifications for a relational database. This step must be performed before the actual data is loaded into the database.

3.5 Developing a Prototype Database

All the accumulated knowledge, expertise, and information are now used to create a prototype database. This database aids in clarifying goals, identifying problems in the design or content, identifying additional resources or information required by the end user, establishing the end users' expectations, and refining the project.

The prototype database should be built from a small subset of the data to be stored in the database. This subset should include at least one example of each of the attributes defined in the schema. If data is obtained from several different sources or serves different end users, the prototype database should contain at least one complete data set from each source and for each end user. If curves or graphs are to be included in the database, they should be included in the prototype to verify presentation. The more realistic and comprehensive the prototype, the more useful it will be.

When the prototype database is available, a group of end users is organized to evaluate it. While each user may not be completely familiar with all the capabilities of the application software, their input will be valuable in determining whether the attributes and database structure are complete and usable. A questionnaire derived from the project requirements can be used to facilitate the evaluation process.

Using the information gained from evaluation of the prototype, the schema is revised, the loading files modified, and the database recompiled. The evaluation, revision, and rebuild cycle is continued until the database is determined to be acceptable. This prototype database should be archived for future reference by moving it to another directory or renaming it.

While this iterative approach of database design, load, evaluate, and re-design may seem time-consuming, it has been proven to be the best method for creating a database that best meets the end users' requirements and is the most efficient method in the long run. The most time-consuming aspect of creating a database usually involves formatting and inputting the actual data. Revising the final database can mean revision of numerous large files.

The final prototype should be subject to a final review before data entry is begun. Designating a review committee and formalizing the procedure will help to ensure a database that meets the acceptance criteria. Final approval should include a consensus of the end users. If the database is too difficult to use or does not provide a real benefit over their current methods, they will continue to use what is familiar to them.

3.6 Populating the Database

The data provider coordinates the effort of formatting the data for entry into the database. Data often comes from disparate sources and is stored in a myriad of media and formats. The process of entering or translating data into the database should include documenting the status of the source data and establishing the most efficient method of loading it into the database

The individual values for each attribute may come from different sources. For each attribute, the data provider should identify:

- Where the source data is located
- How to acquire a usable copy of the source data
- What media it is stored on or will be delivered on
- What format it is stored in and in what format it will be delivered in

To increase the efficiency of the data loading process, it is best to group the data by format and media. Each format and media will have a particular data entry process or method associated with it. Because data can be entered from an unlimited number and type of data files, it is generally easy to accommodate data stored or delivered in different forms.

Data entry of information stored on paper may be simplified by first transferring it to a standard form. Future data acquisition for the same type of data could be entered directly into the same form. This aids in assuring data consistency and completeness. It may also be useful to store some information as documents, which can be accessed in any form or location. Web-enabled applications have greatly increased the usefulness of this method.

For each group of data, the optimum procedure for data entry or transfer of data into the database must be established. There are several ways to load data into a database, largely dependent on the software application:

1. *Load Files.* Load files are typically formatted in ASCII text files but may be any file that is used to populate materials property values into a database. They can be created manually using any word processor or text editor or translated from another electronic format.
2. *Spreadsheets.* Spreadsheet applications are often used to process raw data and to load that data into a database. This method is particularly appropriate for relational databases, which are stored in spreadsheet-like tables.
3. *Manual Entry.* Disparate bits of data or minor revisions can usually be keyed directly into the database using the software application interface.
4. *EXPRESS/XML/MATML.* These standard formats are useful for transferring data from existing software or database applications, if supported by the individual applications.

Having established the best methods for loading data from each data source, populate the database with the material property values required for each end use application.

3.7 Building the Database

Using the best methods for the specified database software application, a final production database is compiled for distribution to the end users. All standard database-building practices should be observed and incorporated. These techniques are unique to the type of database used and are well documented in the database administration literature.

It is important to backup the final database and to control versioning. The security of data sources and the final database should be given appropriate consideration at this point, as these items represent a significant investment.

3.8 Customizing the User Interface

The information gathered in the discovery phase of the database building process can be used to customize the user interface, if required. Most user interface customizations are accomplished with files that are external to the database. Customizations can include creating data-specific reports or creating views (virtual tables) that can be applied globally or on an individual basis. In most cases, these user interface customizations involve queries that are executed to extract subsets of data. Some typical database-related user interface customizations that may have an impact on the database structure, definition, or organization include:

Units Conversion. Database values are generally entered and stored in the units system in which they were measured. Different end-user applications may require a different unit system. The database or user interface software must accommodate this units conversion.

Export/Import Data Mapping and Reports. There are numerous uses for the data extracted from the database. The end use can be simply creating reports for viewing on screen or in hardcopy. Often it involves importing or exporting data to external software programs, such as analysis codes. Data manipulation may be involved during export to transform the database data into the formats required by the external program. The required transformations or mappings may influence the data type, units, or accuracy of values stored in the database.

Database Documentation. It is good practice to provide documentation to the end user that details the database structure and content. This documentation assists users in understanding the database and provides disclaimers or data qualifications.

Indexing Database. The performance of access and query functions is improved significantly by indexing the database. Indexing is not required, but highly recommended.

3.9 Qualifying the Database

The completed database and all associated interface customizations should meet or exceed established acceptance criteria. Quality assurance measures the database against predetermined acceptance criteria and generates feedback to the project team. The project team corrects errors and rebuilds the database. This process is repeated until the finished database and associated interface customizations meet the acceptance criteria. When the database has passed the quality assurance process, the database is submitted for final approval.

Quality assurance should view the database and interface customizations from the end user perspective, while understanding the source data, test methods, etc. If acceptance criteria were not defined as part of the project-planning phase, assemble the project team and carefully define the criteria at the earliest opportunity. Do not wait until the project is complete before defining the requirements.

Every member of the project team can provide input to the quality assurance process. However, it is probably best that the focal point for this activity not be the database builder. Just as it is difficult to edit one's own documents, often others can more easily discover errors in the building process. The quality assurance team is analogous in its functions to the database-building team:

- The project leader provides a focal point for all communications involved in the quality assurance process and coordinates the effort. He reviews the acceptance criteria and finalizes the approval procedures.
- End users verify that the database meets specified requirements. For example, that the database supplies all necessary information in usable format, that the required materials and information for each material is available, that data is easily retrieved in a timely manner, that data transfers accurately to the end-use applications, and that required units conversions are performed.
- The data provider verifies that all specified data was transferred and is stored efficiently and accurately. In addition, he should be available for verification and validation of property.
- The programmer may be required to modify data, translators, mapping files, etc. if the data validation process indicates that data translation or data reduction errors exist.
- The database builder corrects errors in the schema, load files, and customization files, compiles the database, and resubmits it to quality assurance.

Acceptance Criteria. The following database acceptance criteria, specific to materials data management systems, provide a minimum set of standards:

- Does the database contain all the intended materials and data? Verify that attributes have been created and populated for all the properties, materials, and pedigree information requested by each end user to support each end-use application.
- Are commonly used queries included in the user interface? Commonly used queries should be readily available in the user interface. Verify that these queries exist, are intuitive, and return values reliably and in a timely manner.
- Is the data in the database stored correctly? Verify that curve data can be extracted and manipulated, that data is stored in the correct format for the required queries, and that attribute values are within established constraints.
- Can data be exported or transferred to the required end-use applications? Verify that exported values are mapped correctly.
- Are units conversions performed correctly and accurately? Convert units for all attributes in the database; check the returned values and conversion factor for each attribute and each unit system.

4 COMMERCIAL DATABASE MANAGEMENT SYSTEMS

Many manufacturing organizations have programmers on staff, so designing a database management software system in-house is often an option. Cost, schedule, and maintenance issues can quickly eliminate this option. Most commercial DBMS providers can provide volumes of testimony from their customer base regarding the failures of in-house DBMS solutions. “Off-the-shelf” applications, customized to meet the specific database and interface requirements of the organization usually prove to be most cost and time effective.

In-house personnel, using the application programmatic interface (API) supplied with the software, can often readily accomplish customizing a commercial DBMS system. Additionally, most DBMS providers offer customization services as part of an implementation or installation package. Using this approach, in-house personnel can be trained and ready to use the installed DBMS system on site.

Before examining the systems commercially available, it is recommended that you carefully review the requirements for your organization. In addition to storage of in-house data, the following should be considered:

- Whether commercial data is available for use with the DBMS.
- What hardware platforms are available and supported in-house.
- Whether plans exist to link to a PDM system. If not, identify requirements for interfacing between your DBMS and analysis or design software.
- Whether interacting with the supply chain is important to the efficiency of your organization.
- Whether the DBMS will store all required data types.
- Whether you intend to disperse your data locally or globally.
- Whether the DBMS supports materials data standards embraced by your organization and industry.

The following systems were commercially available at the time of this writing. This is not an exhaustive list, but a compilation of several different approaches to the problem of materials data management. Most are supplied by companies that also offer complete implementation services. The following excerpts are derived from product literature.

MSC.Mvision and MSC.Enterprise Mvision, by MSC.Software Corporation. MSC.Mvision is a suite of software applications based on a proprietary database system that is optimized for engineering materials data. Designed and built specifically for engineering applications, they include integrated X-Y graphics, printing, spreadsheet, query language, and web browser capabilities. The applications easily handle complex engineering data types such as numerical values with units, footnotes, numerical precision, images, photographs, and documents.

MSC.Enterprise Mvision provides web-enabled access to MSC.Mvision databases. Using a client-server configuration and a standard web browser, users can view data stored in a single location, eliminating the requirement for local database installations. Designers or engineers can directly search a central knowledge collection, retrieve data, create graphs, export data to analysis codes, or link to external web sites or public-domain databases. Units conversion, footnotes, metadata, and pedigree information is displayed alongside the data values to which they pertain.[13] Additional features of the MSC.Enterprise Mvision Materials Information System include:

- Controlled access to proprietary information.
- Extensive customization and integration options supplement the standard user interface using HTML and Java programming.
- Integrated access from within MSC.Patran and other CAE environments.
- Integrated client capability provides integration with other computer-aided design (CAD) or computer-aided engineering (CAE) software, allowing data search and retrieve capability from within the application.
- An extensive collection of precompiled materials databases from authoritative sources offer over a half million individual materials data records, including a fully queryable version of Mil-Handbook 5.
- Applications are available for use on UNIX, Windows PC, and Linux PC.

CenTOR Software, Inc. CenTOR Software provides business-to-business (B2B) interaction management solutions. Using web-enabled XML-based technology, its software allows customers to optimize business interactions with partners and suppliers. At the core of CenTOR's application suite is the Interactive Server TM, a secure, scalable, and open standards-based platform designed for rapid development, innovation, and cost-effective reuse of ideas information and processes. CenTOR's Design Collaboration product includes the following applications:

- Customer Driven Design enables collaborative modeling of product features and customer requirements, assists in prioritizing engineering activities, leverages the customer voice early in the development process, minimizes value-add features, and measures specific feature impact.
- Materials Identification and Validation enhances materials analysis early in the design process, helps designers leverage preferred suppliers and approved vendor lists, and facilitates collaboration among engineering, manufacturing, purchasing, and supplier organizations.
- Regulatory Compliance Management allows companies to stay current with interchange processes (compliance and conformance) through the aggregation of forms and information for a diverse set of compliance requirements such as Environmental Health and Safety (EH&S) for the Environmental Protection Agency (EPA), Occupational Safety and Hazard Administration (OSHA), and product development for the Food and Drug Administration (FDA).

IDES, Inc. IDES, Inc. provides content conversion and management for materials suppliers, focused primarily on the plastics industry. Utilizing XML technology to capture materials information for dissemination via web browsers, IDES' Content Team assists an organization in bringing product information into one current and comprehensive source for use in e-business initiatives via the Intranet and a Extranet. By incorporating the importance of global test standards and utilizing a multitude of database formats, they provide an effective system for managing technical information.

IDES also has options for manufacturers or original equipment manufacturers (OEMs) who may be implementing ERP systems or other supply-chain solutions. IDES offers customized solutions.

M-Base Engineering + Software, GmbH. The MC-Base material database software application is based on Java technology and is accessible by a web browser. Providing the benefits of centralized access to materials data maintenance and distribution, it makes materials data available to the extended organization. The system offers search and sort capabilities, single-point and multipoint data storage, graphics features, and complete data maintenance and administration tools.

The components of this system include an Internet browser with Java 2 support, Oracle Application Server, and an Oracle Database System. M-Base provides programming expertise for modification to their software to integrate existing in-house software and CAE programs.

Cambridge Materials Selector (CES3), Granta Design Limited. CES3 is designed to manage materials and manufacturing process information within an organization, providing cost savings, superior design capability, preferred list management, and fast information flow. CES3 incorporates the "design-led" selection methods of Prof. Mike Ashby of Cambridge University, allowing engineers to determine optimal choices of materials and processes. There are six main parts to the CES3 system:

- Selector, the latest implementation of the intuitive graphical method for selection pioneered by Ashby and co-workers at Cambridge University Engineering Department.
- CES Data Modules, comprised of high-quality, validated data—materials, manufacturing processes, structural sections, suppliers, references, uses, and industrial sectors.
- Constructor, the database development kit for CES3, making the system customizable and extensible.
- InDepth, providing background information about the CES system—the selection methodology, the databases, reference information, and 50 fully worked interactive case studies.
- Viewer and Weblinks, providing access to high-quality information on CD-ROM and the Internet, respectively.

CES3 is intended principally for four application areas:

- Decision support for engineers with demanding design requirements
- Management of in-house company information relating to materials, processes, shapes, standard components, or other engineering knowledge bases
- Marketing by manufacturers and distributors of materials, process equipment, structural sections, or standard components
- Education of materials selection in engineering design, process selection in manufacturing, and other related subjects

This product requires a PC with a Pentium processor, using Windows 95 or Windows NT4 or above.

MAPP, by Matec Materials Technology Software and Data Services. MAPP is a database management system with an adaptable, open structured interface and search engine. It was designed specifically to enable materials scientists and engineers to develop their own materials database under Windows or for use on Apple Macintosh systems. It can be used to:

- Construct a database containing materials by trade and common name, composition, specification, and property data.
- Search for materials by any of the above entry criteria; list materials meeting maximum and minimum values for one or more properties.
- Export data to spreadsheet, word processing, or graphics programs.

5 MATERIALS DATA STANDARDS

Materials data compilations represent a considerable investment and, therefore, usage must be maximized. When the development of a materials database system for the dissemination of materials data, either within or outside of an organization, is considered in conjunction with the requirements for integration with other systems, the need for standardized representation of materials data becomes obvious.

There are two distinct types of standards for materials data: standards for materials test data reporting and standards for materials data exchange, including those for transfer of data in electronic format to CAD/CAM/CAE systems. Organizations active in writing standards and coordinating development for materials test data reporting are:

- ISO International Organization for Standardization (TC 61 SC 13 WG 1-6)
- NIST Office of Standard Reference Data
- DoD Technology Standards Area (Mil-Handbook 17, Mil-Handbook 5)
- ASTM Standards for Composites Materials (D20, D30, C28)

Organizations active in materials data exchange standards are:

- NIST Center for Manufacturing Technology
- ASTM Computerization of Material Property Data
- NIST Institute for Computer Science and Technology
- ACC Automotive Composites Consortium

The following summarizes the evolution of the standards most commonly used by the materials community for the standardized reporting and exchange of engineering materials data.

IGES/PDES. The IGES/PDES Organization was coordinated in the late 1970s from industry, government, and academia to develop standards and technology for the exchange of product information between different CAD systems.[16] This group focused its efforts on two projects, the Initial Graphics Exchange Specification (IGES) and Product Data Exchange Specification (PDES) using STEP. This effort resulted in the publication of IGES in 1980, which was subsequently adopted as an ANSI standard. IGES Version 5.3 was published in 1996.

A second-generation Product Data Exchange (PDE) technology, Product Data Exchange Specification (PDES), was initiated during the mid-1980s and was submitted to ISO in 1988. The international community adopted it as the basis for ISO 10303 (STEP).

Today, the ongoing PDE technology efforts include the primary Product Data Exchange using STEP (PDES), an American National Standard (ANS). This project is the primary U.S. project providing industry inputs to this ISO activity. Fourteen international standards have been created as a result of this effort. More than 20 countries worldwide have approved STEP, including all major U.S. trading partners.

The Initial Graphics Exchange Specification (IGES) defines a neutral data format that allows for the digital exchange of information among computer-aided design (CAD) systems. CAD systems are in use today in all phases of the design, analysis, manufacture, and testing of products. Since it is common practice for a designer to use one supplier's CAD system and for the contractor and subcontractors to use different systems, there is a need to exchange data among CAD systems. Using IGES, a user can exchange data models in the form of wire frame or solid geometry representations. Applications supported by IGES include traditional engineering drawings as well as models for analysis and/or various manufacturing functions. In addition to the general specification, IGES includes application protocols in which the standard is interpreted to meet discipline-specific requirements.

Version 1.0 of the specification was adopted as an American National Standard ANS Y14.26M-1981 in November of 1981. The current version, IGES 5.3, was approved by ANSI under the new guidelines of the U.S. Product Data Association (US PRO) during September 1996. Under the latest distribution agreements with ANSI, US PRO has obtained permission to distribute IGES 5.3 in both paper and digital (e.g., CD-ROM) formats. The latest version of the IGES standard is designated ANS US PRO/IPO-100-1996.

IGES version 6.0 is slated to be the next, and final, release of the standard. The IGES project has posted a list of the Edit Change Order extensions approved for 6.0. Current plans call for the support of activities related to maintaining the existing IGES capabilities with all new requirements being forwarded for consideration in the appropriate parts of the STEP/PDES standards.

ASTM Committee E 49. The ASTM Committee E 49 is responsible for one of the leading efforts in materials property data representation in computerized systems. The various subcommittees specialized in areas such as materials designation, data recording formats, terminology, data exchange, and data and database quality. The compiled set of standards is included in Volume 14.1 of the *Annual Book of ASTM Standards.*[17]

ISO/STEP. The most comprehensive effort toward standardization was the development of STEP in 1995 (formally ISO 10303; Standard for Product Data Representation and Exchange), which was designed to model all aspects of a component throughout the life cycle of the product. STEP uses a data-modeling language known as EXPRESS for its neutral format, which while originally created to facilitate automatic generation and interpretation of computer software, is also readable in ASCII format. Within this model are all the tools required for efficient data transfer, known as parts. Specifications called application protocols (APs) are used to apply the general concepts outlined in the parts to particular classes of products.[18] Through the implementation of APs, STEP is extensible across a wide variety of applications and industries.

The Materials Model, Part 45, defines a material as a manufactured object with associated properties in the context of its use environment.[19] Unlike traditional textbook views of materials with limit point properties abstracted from microscopic representative volume elements, Part 45 represents the actual property distributions in a finite volume material object as manufactured. This more robust definition is required to design robust products. The textbook definition is adequate for conceptual design (screening) and is a subset of the Part 45 definition.

The standards required to integrate the materials property data into the CAD/CAE environment require math forms to represent linear or nonlinear materials properties. Linear property models for finite element mechanical and thermal analysis are included in Part 104 and are incorporated into AP 209 for modeling metallic and composite structures. Other applicable parts and application protocols include:

- Part 42—Geometric and Topological Representation
- AP 203—Configuration Controlled Design
- AP 214—Core Data for Automotive Design Processes

The advantages of using such a comprehensive standard as STEP in a materials data management system include[20]:

- Ready-made data structures that are rich in detail
- The integration of dispersed data systems
- Flexibility for different purposes
- Automatic integration with different CAE systems

- More complete audit trails for the origins of data
- Increased quality assurance
- Reduced costs, liability, and risk

Finally, at the end of a product life cycle, ISO 14000 compliance is required as regulatory agencies become increasingly strict about environmental regulations.

In addition to the current international standards, work is underway on over 30 additional parts of STEP.

Materials Consortia. Various collaborative consortia have developed their own standards for modeling materials data, which have become *de facto* standards for the respective industry. These consortia implemented a common technology, data structure, or modeling format for the data stored in their databases. The resulting populated databases represent a competitive advantage and are generally confidential to members of the consortia. Some examples include electronic packaging materials, carbon–carbon materials for rocket engines, and creep data on steels used in the power station industry. Noteworthy efforts include:

- The Mil-Handbook 17 committee documents the standards for storage of composites test data used by leading aerospace organizations.[21] The Mil-Handbook 17 schema provides a structure for over 500 attributes of composites test results and processing history of the materials to which the data pertain, compliant with Mil-Handbook 17 terminology.
- The Automotive Composites Consortium and ASTM Committee E-49 collaborated to develop standards for the computerization of composite materials and metadata.[22] This early work provided guidelines for composites data storage, identifying the importance of metadata (also referred to as pedigree) in defining the material. Over 50 parameters are required to adequately represent a composites material test specimen. These properties serve as the basis for an audit trail when the source or history of a property is required.[23]
- The Computer-Aided Materials Preselection by Uniforms Standards (CAMPUS R) consortium of plastics suppliers has developed a database used worldwide for storing materials information on plastics. Provided for use on a PC, it uses a uniform database structure and interface for all participating suppliers, allowing screening of materials for new applications. All properties included in the CAMPUS database are tested to one of two international standards, ISO 10350 (single-point data) and ISO 11403 (multipoint data), parts 1 and 2, for comparable data.

Materials Data Standardization and the Web—MATML/XML. When considering computerized access to materials data, one must start by recognizing that hundreds of different engineering material designation schemas are used by trade organizations, standards agencies, government agencies, and individual companies. Virtually all were developed prior to the era of web-connected computers. Therefore, a natural language capability is required to search the universe of material databases. There are no absolute standard designations available for engineering materials. Therefore, even when the same database software system is used to build a set of databases, web searches are difficult due to the different designation systems required for different material types.[9]

The dissemination and exchange of information on the web is typically performed using HTML (hypertext markup language). HTML, designed to describe a document's form rather than content, does provide flexibility for those in a given industry to establish their own language for their own purposes, meaning HTML is not extensible. Communities of web users rapidly reached the point where they wanted to move beyond the dissemination of information into the exchange of information.[24]

Recognizing these shortcomings, the World Wide Web Consortium (W3C) embarked on an effort to bring an abridged implementation of the standard generalized markup language (SGML), a language used to describe languages, to the web. The result of the W3C's effort was the February 1998 release of the version 1.0 specification of XML, the extensible markup language. XML provides:

- Extensibility, so users can define their own tags and attributes used in their documents.
- Structure, so users can define their own document-type definition (DTD), the information model for a document describing how the tags and attributes are combined.
- Validation, so users can test the conformance of their documents to the structure defined by the DTD.

Additionally, XML was designed to be easy to learn, easy to write, easy to interpret, and easy to implement; characteristics perfectly suited for use on the web. Many excellent sources of information are available on XML on the web, such as the W3C's XML pages.

There are now many applications of XML in use by different communities. Two well-known examples in the sciences are MathML, developed by mathematicians to represent mathematical notation and CML, the chemical markup language, designed to manage chemical information. In October 1999, a new effort called MatML was launched to develop an extensible markup language for materials property data. MatML permits the storage, transmission, and processing of materials property data distributed via the world wide web. The MatML Working Group, coordinated by the National Institute of Standards and Technology, represents a cross section of the materials community with members from private industry, government laboratories, universities, standards organizations, and professional societies, whose strategy includes dissemination of MatML through a variety of standards and professional organizations.

The Working Group has recently produced a working draft of the document type definition (DTD) for the markup language. The MatML DTD contains structures for transferring information concerning the material and its properties and terms for its interpretation. The DTD is the XML semantic and syntactic formalism that software will need to parse, interpret, and use the data contained in MatML documents. The MatML DTD is soon to be subjected to application development and acceptance testing, which will be crucial in demonstrating that MatML works, testing its robustness, and serving as a guide for others interested in using MatML.[16] Data encoded in MatML can be utilized by web browser components, import filters to spreadsheet and database programs, import filters to CAE software, parsers, and editors.

6 SUMMARY

A materials data management system can significantly improve the efficiency and profitability of an organization when strategically integrated into an organization. Its success depends upon the full commitment of management, an implementation that supports the company's business objectives, and careful planning to provide access to the required data for each end use function throughout the product life cycle. Optimized use of commercially available systems, customized to meet the functional and integration requirements of the organization, can help to ensure a positive return on investment. Utilization of recognized database management practices and the increased ease of data transfer between software applications offered by the use of established industry standards can help to create a system that is user friendly and easily adopted by the entire organization.

Acknowledgments

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3. Terry Wong, "Optimizing the Engineering Process at Rocketdyne Using MSC/MVISION," Boeing North America, Inc., Rocketdyne Division, Canoga Park, CA, 2000, p. 4.
4. Robert Mills and Lisa Kember, "PDM Comes on Strong," *Computer Aided Engineering News,* May (1996).

5. Ken Blakely, Larry Johnson, Boma Koko, Ray Amador, and Arthur H. Fairfull, “Integrating CAE and PDM: A First Step Toward Providing Simulation Data Management,” *Integrated Enterprise,* Spring 2001.
6. Ed Miller, “Web Technology Comes to PDM,” *Computer-Aided Engineering News,* May (1966).
7. Michael Hammer, “Out of the Box: The Internet’s True Benefit the Net’s Real Payoff Is in Its Enabling of Business Collaboration,” *Informationweek.com,* August 21, 2000.
8. Arthur H. Fairfull, “Integration of Materials Data in Computer Aided Engineering,” MacNeal-Schwendler Company, Ltd., 2000.
9. Edward Stanton, Thomas Kipp, and Eric Lantz, “Recent Developments in Materials Data Management,” The MacNeal-Schwendler Corporation, Costa Mesa, CA, p. 5.
10. J. E. Lee, D. E. Marinaro, M. E. Funkhouser, R. M. Horn, and R. R. Jewett, “Creating a Common Materials Database,” *Advanced Materials and Processes,* ASM, OH, November (1992) reprint.
11. Chris Kirtley, “Modeling Interactively with the Materials Database,” *Materials Database News,* Vol. 2, GEC Alsthom, MEC Whetstone, August (1996).
12. R. G. G. Cattell, *Object Data Management: Object-Oriented and Extended Relational Database Systems,* Addison-Wesley, 1991, pp. 48–52
13. Arthur Fairfull, “MSC Enterprise Mvision: Materials Property Data on the Web, Supporting Engineering Simulation,” *Integrated Enterprise,* Spring, 25–28 (2001).
14. http://www.ceramics.nist.gov/srd/summary/aboutadv.htm.
15. Michael J. Hernandez, *Database Design for Mere Mortals,* Addison-Wesley, 1997, pp. 273–274.
16. http://www.nist.gov/iges, copyright © 1997–2001 US PRO.
17. *Computerized Systems; Computerized Material Property Databases,* Vol. 14.01, 1994 *Annual Book of ASTM Standards,* ASTM PCN 01-140194-32, ASTM, West Conshohocken, PA, 1994.
18. “The Development of an Application Protocol for the Representation and Exchange of Data from Engineering Plastics,” Ferroday Ltd, England, 1993.
19. Normal Swindolls (Ed.), ISO 10303 Part 45, Materials, ISO TC184/SC4.
20. “Computerized Materials-Information Systems,” *Phil. Trans. Royal Society London A,* **322,** 373–391 (1987).
21. R. Fields (Ed.), “Database Schema Development Committee for Composites, Composite Material Test Data Schema Version 1.0,” 1996-02-02, Information by T. Kipp, MSC.Software Corporation, Santa Ana, CA, 1996.
22. Edward L. Stanton, “Computerization of Composite Materials Data and Metadata,” PDA Engineering, Costa Mesa, CA, 1989.
23. N. Swindolls, “Materials Property Data in STEP,” Proceedings of the Data Conference, September 1994.
24. E. F. Begley, NIST, July 2000, http://www.ceramics.nist.gov/matml/about.htm.

CHAPTER 18

INFORMATION FOR MATERIALS PROCUREMENT AND DISPOSAL

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Ballston Spa, New York

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3 DIRECTORIES AND CROSS REFERENCES TO MATERIALS STANDARDS AND SPECIFICATIONS 511
4 MATERIALS DISPOSAL 513
5 SOURCES OF INFORMATION FOR MATERIALS DISPOSAL AND RECYCLING 514
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1 INTRODUCTION

No article can be produced in today's economy without access to substantial procurement information on the constituent materials specified by engineering for its manufacture. Furthermore, today's demands for environmental protection and conservation require materials information of other sorts, both for disposal of the article itself and for the scrap generated during its manufacture. The needs and sources for these types of information are summarized in Fig. 1, which displays the role of materials information throughout the design–manufacture–use cycle. In this chapter we will detail the kinds of information needed for materials procurement and disposal and summarize the available sources for this information. Finally we review some current issues affecting materials procurement–globalization, e-commerce, and "just-in-time" logistics management—the function of information in optimizing the business impact of each issue, and the consequent new demands on information—currency, computer access, etc.

2 PROCUREMENT INFORMATION

Designation and Specification. The task of materials procurement in an industrial enterprise cannot be likened to that of a grocery shopper who makes a simple list (apples, bread, eggs, etc.), drives off to the local supermarket, and makes acquisitions by visual inspection and on-the-spot price comparison. In contrast, the industrial purchasing agent must first know how is this material

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Handbook of Materials Selection. Edited by Myer Kutz

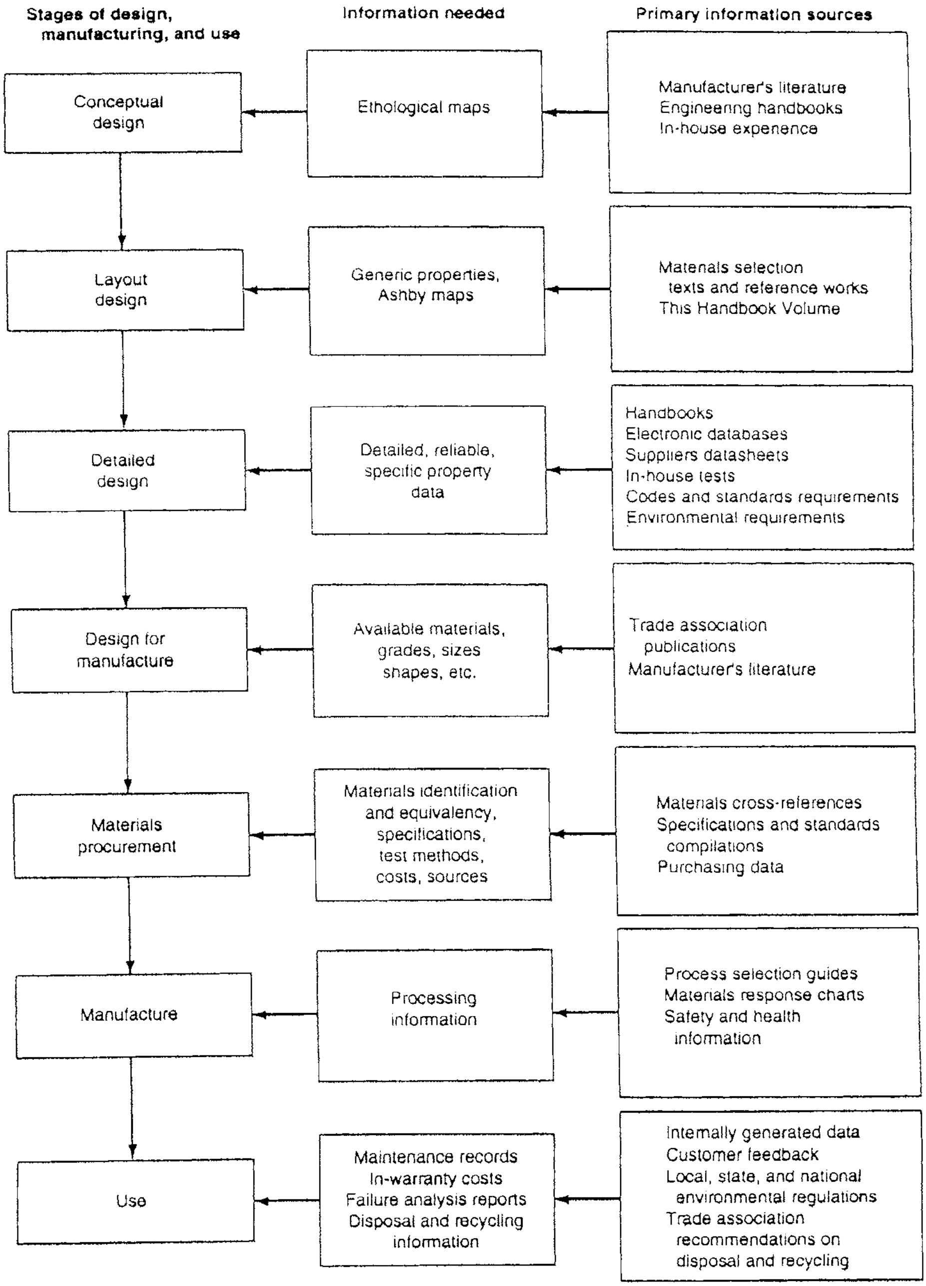


Fig. 1 Information needs and sources at various steps in the design–manufacture–use cycle.[4]

identified, who are the leading producers and their addresses, in what unit quantities is it sold, to what specifications or standards must it conform, etc. To answer the first question, the agent must know the most common designation for the material—international, national, trade association, or other—and its equivalence in other designation systems. Various directories and cross-reference guides have been developed for this purpose, a summary of which is provided in Section 3. A typical example of a family of equivalents is shown in Table 1.

Nominal equivalence, as set forth in a cross-referencing table or by chemical composition as in Table 1, may not suffice. Thus any material offered by a prospective supplier, whose specification is not explicitly that called for by the design engineer, should be tested for critical properties and a sign-off obtained from engineering prior to purchase. The sources cited in Section 3 are primarily cross-reference guides and seldom offer more than a brief summary of a specification. Therefore, to assist the engineer in determining the equivalence or acceptability of the material in question, resort must be made to the full specification for the material. These may be obtained from the issuing body—International Organization for Standards (ISO), American National Standards Institute (ANSI), American Society for Testing and Materials (ASTM), Deutsches Institut für Normung (DIN), British Standards Organization (BSO), etc. Conveniently, a very comprehensive collection of standards and specifications of all types is maintained by Information Handling Services, Englewood, Colorado, which can provide printed copies of any selected specification for a nominal charge.

The purchasing agent should work with the engineer to secure the most cost-effective material. In particular, effort should be made by a company to avoid "specials" and to try whenever possible to adopt a standard or specification already established by an organization at a higher hierarchical level, that is, a trade association, professional association, or national or international standard. Whenever necessary to be included in a specification, the test method or quantitative inspection technique cited should be one recognized nationally, for example, an ASTM standard. The subject of standards and specifications for materials has been discussed by Westbrook.[2]

Materials Sources. Having identified the material needed and the specification(s) governing its properties, the purchasing agent must consider possible sources from which it might be obtained. These should include both sources local to the plant of the procuring organization, national sources, and international sources. Whether or not a given vendor is a viable source will depend on stocking policies, delivery schedules, price, etc. Economic aspects are discussed below under costs. General guides to materials sourcing are as follows:

Sources of Materials Purchasing Information

American Metal Market (5 issues per week) Fairchild, NY

Basil, D. C., et al., eds., *Purchasing Information Sources*, Gale Research, Detroit MI, 1977, 256 pp.

Bittence, J. C., ed., *Guide to Engineering Materials Producers*, ASM International, Metals Park, OH, 1993, 392 pp.

Brady, G. S., Clauser, H. R., and Vaccari, J. A., *Materials Handbook: An Encyclopedia for Managers, Technical Professionals, Purchasing and Pro-*

Table 1 Example of Chemical Composition Comparison of U.S., International, and Foreign Specifications for a Given Material (305 Stainless Steel)[1]

Nation	Agency	Designation (Alternate)*	C	Si	Mn	Cr	Ni	Mo	Others
USA	UNS	S30500 (AISI 305)	0.12	1.00	2.00	17.00–19.00	10.00–13.00	—	
International	ISO	683 / XIII Type 13	0.10	1.00	2.00	17.00–19.00	11.00–13.00		
USA Society	AMS	5514 (*p, sh, st*)	0.12	1.00	2.00	17.00–19.00	10.00–13.00	0.75	0.75 Cu
USA Society	AMS	5685 (*w*); 5686 (*w*)	0.08	1.00	2.00	17.00–19.00	10.00–13.00	0.75	0.75 Cu
USA Society	ASTM	A193 (*bolt*), A320 (*pipe*), A240 (*p, sh, st*), A276 (*b, shp*), A313 (*w*), A314 (*b, bl*), A368 (*w*), A473 (*f*), A478 (*w*), A492 (*w*), A493 (*b, w*), A511 (*t*), A554 (*t*), A580 (*w*) UNS S30500	0.12	1.00	2.00	17.00–19.00	10.50–13.00		
USA Society	SAE	J405 30305	0.12	1.00	2.00	17.00–19.00	10.50–13.00	—	
USA Government	FEDERAL	QQ-S-763 305 (*b, f, w*)	0.12	1.00	2.00	17.00–19.00	10.00–13.00	0.75	0.75 Cu
USA Government	FEDERAL	QQ-S-766 305 (*p, sh, st*)	0.12	1.00	2.00	17.00–min	10.00–13.00	—	
USA Company	GE-EMPIS	B7A39 (*w*); B7A41 (*sh, st*)	0.12	1.00	2.00	17.00–19.00	10.00–13.00	—	
Bulgaria	BDS	9631 Ch18N9L	0.15	1.50	2.00	17.00–19.00	8.00–11.00	—	
France	AFNOR NF	A35-575, -577; ZBCN18.12; A35-586 305F00	0.10	1.00	2.00	17.00–19.00	11.00–13.00	—	
France	AFNOR NF	Z10CN18.9M	0.12	2.00	1.50	17.00–19.50	8.00–10.00	—	
Germany, Dem R.	TGL	14394 GS-X12CrNi18, 9	0.15	1.50	2.00	17.00–19.00	8.00–10.00	—	
Germany, Fed R.	DIN Stoff Nr	1.4303,X5CrNi18 12 (X5CrNi19, 11)	0.07	1.00	2.00	17.00–19.00	11.00–13.00	—	
Germany, Fed R.	DIN Stoff Nr	1.4312, G-X10CrNi18, 8	0.12	2.00	1.50	17.00–19.50	8.00–10.00	—	
Italy	UNI	AIP XBCrNi19, 10	0.08	1.50	1.00	18.00 nom.	12.00 nom.	—	
Japan	JIS	G4303; G4304; G4305; G4308; G4309; G4315; G4318 SUS305 (SUS62HP, CP, WR)	0.12	1.00	2.00	17.00–19.00	10.50–13.00	—	
Japan	JIS	G4308; G4309; G4315; G4318 SUS305J1	0.08	1.00	2.00	16.50–19.00	11.00–13.50	—	
Mexico	DGN	B-171 Grade MT305; B-218 Grade TP305	0.12	1.00	2.00	17.00–19.00	10.00–13.00	—	
Mexico	DGN	B-83 305	0.12	1.00	2.00	17.00–19.00	10.50–13.00	—	
PRC China	GB	1220 1Cr18Ni12; 3280 1Cr18Ni12	0.12	1.00	2.00	17.00–19.00	10.50–13.00	—	
				1.00					
Rumania	STAS	6855 T12MSNC180	0.15	0.50–0.20	2.00	17.00–19.00	8.00–10.00	—	
Spain	UNE	36-016 F.3513 XBCrNi18-12 E-305	0.10	1.00	2.00	17.00–19.00	11.00–13.00	—	
United Kingdom	B.S.	1449 305S19	0.10	1.00	2.00	17.00–19.00	11.00–13.00	—	
United Kingdom	B.S.	3111 305S17	0.07	1.00	2.00	17.00–19.00	11.00–13.00	—	
USSR Russia	GOST	06Ch18N11	0.06	0.80	2.00	17.00–19.00	10.00–12.00	.30	0.30 Cu, 0.20 W, 0.20 Ti
USSR Russia	GOST	10Ch18N9L	0.07–0.14	0.20–1.00	1.00–2.00	17.00–20.00	8.00–11.00	—	0.30 Cu

* *b* = bar, *bl* = billet, *f* = forging, *p* = plate, *sh* = sheet, *shp* = shape, *st* = strip, *t* = tube, *w* = wire.

duction Managers, Technicians, and Supervisors, 14th ed., McGraw-Hill, New York, 1996, 1072 pp.

Cavinato, J. L. and Kauffman, R. G., eds., *The Purchasing Handbook*, 6th ed. McGraw-Hill, New York, 1999, 1082 pp.

Ceramic Sources (annual), American Ceramic Society, Westerville, OH

Chemical Economics Handbook, Stanford Research Institute, Palo Alto, CA, 1982–1984

Cost Reduction Strategies, NAPM Resource Guide, NAPM, P.O. Box 22160, Tempe, AZ 85285-2160 (NAPM is now the Institute for Supply Management)

Frick, J., ed., *Woldman's Engineering Alloys,* 9th ed., ASM International Metals Park, OH, 2001, 1400 pp.

Global Sourcing, NAPM Resource Guide, NAPM, P.O. Box 22160, Tempe, AZ 85285-2160 (NAPM is now the Institute for Supply Management)

Grant, A. H., *A Guide to Buying Castings*, State Mutual, NY

Heinritz, S. F., et al., eds., *Purchasing: Principles and Applications*, 8th ed., Prentice Hall, Upper Saddle River, NJ, 1991, 580 pp.

Leenders, M. R. and Fearon, H. E., eds., *Purchasing and Materials Management*, 10th ed., Irwin, Homewood, NJ, 1992

Locke, D., *Global Supply Management: A Guide to International Purchasing*, McGraw-Hill, New York, 1996, 300 pp.

Product Sources (annual), R&D Magazine, Cahners, Newton, MA

National Association of Purchasing Managers (now the Institute for Supply Management), *Suppliers Directory*, NAPM P.O. 22160, Tempe, AZ 85285-2160

Roskill's Metals Databook, 7th ed., TMS-AIME, Prices, production and producers of 32 metals worldwide

Roskill Reports. Prices, trends, demand, uses, etc. for 70 commodities (metals, minerals, inorganics, bromine, etc.) TMS-AIME, updated regularly

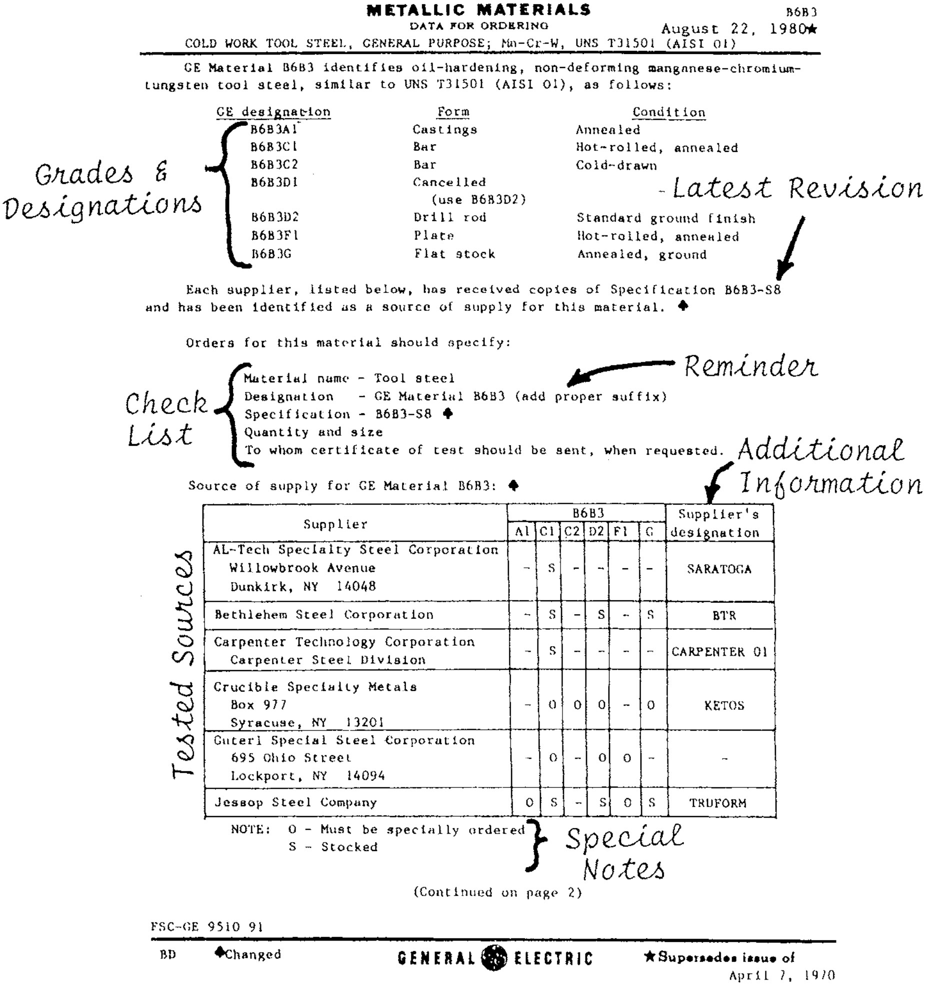
Serjeanson, R., ed., *Metal Bulletin's Prices and Data*, 6th ed., Metal Bulletin Journals, Ltd., Worcester Park, UK, 1982, 423 pp.

Westbrook, J. H. and Reynard, K., "Data Sources for Materials Economics, Policy, and Management," in *Concise Encyclopedia of Materials Economics, Policy, and Management*, M. B. Bever, ed., Pergamon Press, 1993, pp. 35–43

Costs. Actual costs of a procured material are determined by interaction between the purchasing agent and the supplying vendor. These are not necessarily fixed and neatly listed in the supplier's catalog but may depend on a number of factors of which the purchaser should be aware. These include: Is the material regularly stocked or only available on special order; is price affected by size, shape, or surface finish; are there quantity discounts or order quantity minima; what are the normal delivery schedules; are there particular features required that the vendor company would regard as "specials"; etc. General information about material costs and price trends may be found in the sources given above, particularly, *American Metal Market, Chemical Economics Handbook,* the *Roskill Metals Databook* and *Reports,* and the Serjeanson book.

One Company's Reduction to Practice. For more than 70 years the General Electric Company has had a formalized, comprehensive, reference system for the materials information needed in its diverse manufacturing operations. This system is known as EMPIS, an acronym for Engineering Materials and Processes Information Service, and is organized in a tripartite structure, sometimes referred to as the three legs of the materials information stool: engineering properties, specifications, and data for ordering. A fuller description is provided by Westbrook.[3] The part that is relevant here is the Data for Ordering section.

A typical page for a single material among the more than 25,000 materials covered in GE's EMPIS system is shown in Fig. 2. In addition to the EMPIS material number, title, and scope and designation sections, which are similar to



METALLIC MATERIALS B6B3

DATA FOR ORDERING August 22, 1980★

COLD WORK TOOL STEEL, GENERAL PURPOSE; Mn-Cr-W, UNS T31501 (AISI O1)

GE Material B6B3 identifies oil-hardening, non-deforming manganese-chromium-tungsten tool steel, similar to UNS T31501 (AISI O1), as follows:

GE designation	Form	Condition
B6B3A1	Castings	Annealed
B6B3C1	Bar	Hot-rolled, annealed
B6B3C2	Bar	Cold-drawn
B6B3D1	Cancelled (use B6B3D2)	
B6B3D2	Drill rod	Standard ground finish
B6B3F1	Plate	Hot-rolled, annealed
B6B3G	Flat stock	Annealed, ground

Grades & Designations

Latest Revision

Each supplier, listed below, has received copies of Specification B6B3-S8 and has been identified as a source of supply for this material. ♣

Orders for this material should specify:

Material name - Tool steel
Designation - GE Material B6B3 (add proper suffix)
Specification - B6B3-S8 ♣
Quantity and size
To whom certificate of test should be sent, when requested.

Check List

Reminder

Additional Information

Source of supply for GE Material B6B3: ♣

Supplier	B6B3						Supplier's designation
	A1	C1	C2	D2	F1	G	
AL-Tech Specialty Steel Corporation Willowbrook Avenue Dunkirk, NY 14048	-	S	-	-	-	-	SARATOGA
Bethlehem Steel Corporation	-	S	-	S	-	S	BTR
Carpenter Technology Corporation Carpenter Steel Division	-	S	-	-	-	-	CARPENTER 01
Crucible Specialty Metals Box 977 Syracuse, NY 13201	-	O	O	O	-	O	KETOS
Guterl Special Steel Corporation 695 Ohio Street Lockport, NY 14094	-	O	-	O	O	-	-
Jessop Steel Company	O	S	-	S	O	S	TRUFORM

Tested Sources

NOTE: O - Must be specially ordered
S - Stocked

Special Notes

(Continued on page 2)

FSC-GE 9510 91

BD ♣Changed GENERAL ELECTRIC ★Supersedes issue of April 7, 1970

Fig. 2 Typical data for ordering sheet.[3]

the corresponding sections of the specification document, the Data for Ordering sheet contains a checklist, a reference to applicable documents, a list of tested sources of supply for each of the various grades covered by the specification, and when applicable the appropriate supplier's designation for the product. (The supplier's designations are not a part of the specification but are included on the Data for Ordering sheet as clarifying information).

A checklist is provided that tells the buyer at a glance exactly what must be written on the purchase order and what documents are to be included with the order to completely define the desired material. The list of suppliers provided includes only those that have previously delivered conforming material to the General Electric Company and are added to the list at the request of operating components of the company that testify to compliance. To this extent they are treated as sources of supply. However, the list is not to be construed as an ‘‘approved'' list of suppliers, nor as an all-inclusive list. Buyers are encouraged to develop additional sources of supply—particularly for critical or large volume applications—and to report satisfactory experience for subsequent addition to the list of suppliers. Each supplier listed on the Data for Ordering sheet is placed on an automatic distribution list and receives updated copies of each specification for which the supplier is listed. Therefore, when buying from a listed supplier, it is not normally required that a copy of the specification accompany the order.

In many cases the Data for Ordering sheet contains special notes, hints, and suggestions for facilitating procurement, minimizing costs, and standardizing sizes and lots. These are of many different kinds but include, for example, sizes stocked by listed suppliers, cost information, minimum order quantities, delivery schedules, features that represent ‘‘specials,'' etc.

3 DIRECTORIES AND CROSS REFERENCES TO MATERIALS STANDARDS AND SPECIFICATIONS*

(C) *Alloy Finder CD-ROM*, 2nd ed., ASM International, Metals Park, OH. Incorporates *Woldman's Engineering Alloys*, 8th ed., 1994; *Worldwide Guide to Equivalent Irons and Steels*, 3rd ed., 1993; and *Worldwide Guide to Equivalent Nonferrous Metals and Alloys*, 3rd ed., 1996.

(P/C) *Worldwide Guide to Equivalent Irons and Steels*, 4th ed., ASM International, Metals Park, OH, 2000. Available in print (1132 pp.) and on CD-ROM; data from a prior edition also included in *Alloy Finder CD-ROM* (see separate listing).

(P/C) *Worldwide Guide to Equivalent Nonferrous metals and Alloys*, 3rd ed., W. Mack, ed., ASM International, Metals Park, OH, 1995. Available in print and on diskette; data also included in *Alloy Finder CD-ROM* (see separate listing).

(P) Frick, J. P. ed., *Woldman's Engineering Alloys*, 9th ed., ASM International, Metals Park, OH, 2001. Lists compositions, selected properties, and man-

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* C = computer-readable, P = print, P/C = both.

ufacturers for 53,000 alloys. Available in print and on diskette; data from prior edition also included in Alloy Finder CD-ROM (see separate listing).

(P) *British and Foreign Specifications for Steel Castings*, Steel Casting Research and Trade Association, Sheffield, UK, 1980.

(P) *Department of Defense Index of Specifications and Standards*, Part 1, Alphabetical Listing; Part 2, Numerical Listing, U.S. GPO (annual).

(C) *EQUIST for Windows*, M. Gorglay, ed. Hungarian database of standard steels with comparisons and equivalencies for 13,000 standard steels from 18 countries, 1998.

(P) *Index of Standards*, European Committee for Iron and Steel Standardization (ECISS), PSP2, British Steel, plc, Head Office Standards, London, UK 1986.

(P) *German Standards*, Beuth, Germany. English translation of more than 4750 popular DIN standards plus 1500 DIN-EN, DIN-EC, and DIN-ISO standards.

(C) *Global Standards Network.* A directory on the World Wide Web of >100,000 regional, national, and international standards, produced by ANSI and NIST (http://www.nssn.org).

(P) *Handbook of Comparative World Steel Standards*, DS 67, ASTM, 1996, 552 pp.

(P) *Handbook of International Alloy Compositions and Designations*, Vol. 1R, Titanium, Metals and Ceramics Information Center, Battelle Columbus Laboratories, 1990.

(P) Hufnagel, W., ed., *Key to Aluminium,* 4th ed., Aluminium AG, Dusseldorf, Germany, 1991.

(P) *SAE Aerospace Standards Index, Society of Automotive Engineers, 2000.*

(P) *Iron and Steel Specifications,* 7th ed., British Steel, London, UK, 1989, 346 pp. Steel grades to BS970 together with French, German, Japanese, and Swedish standards.

(P) *ISR/VAMAS Unified Classification Scheme for Advanced Ceramics,* ASTM, 1993.

(P) Kehler, M., *Aluminium,* Vol. 3, *Handbook of International Alloy Compositions and Designations,* Aluminium Verlag, Heyden, Germany, 1981, 859 pp.

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4 MATERIALS DISPOSAL

Proper disposal and recycling of materials serves several different objectives: conservation of resources, reduction of energy consumption, lowered materials costs, and regulatory compliance [local, state, and national, e.g., Environmental

Protection Agency (EPA) regulations]. Specific instances include the separation of valuables, e.g., platinum from automotive emission controls, and the separation of hazardous or deleterious contaminating elements, e.g. Pb, Hg, Sn, and Zn. Materials scrap is distinguished as to that resulting from obsolete or worn-out product (obsolete scrap) and that internally generated during manufacturing (prompt scrap), the latter being further broken down into that which is immediately internally recycled (home scrap) and that which is input to the scrap recycling industry (purchased scrap). Throughout all these movements of potentially useful material, information is needed as to the form, composition, and source of the scrap.

Some industries even produce scrap specifications; the iron and steel industry alone has more than 20 such documents covering different grades of ferrous scrap. To realize the highest value from scrap recovery and recycling, or to avoid penalties from improper disposal, thorough identification is needed. This in turn requires reference information on physical properties (color, density, hardness, magnetic behavior, thermoelectric properties, spark characteristics, etc.) and chemical properties (qualitative spot tests, quantitative chemical analyses, or optical and X-ray spectroscopies). If the scrap cannot be explicitly defined as to the particular material (alloy, solution, blend, etc.), then it will be used in a downgrading operation, with lowered cost recovery and heightened possibility of contamination. Some particular sources of information on materials disposal and recycling are listed in Section 5. Other may be found in Refs. 4 and 5.

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6 CURRENT ISSUES

In Section 1 we mentioned some current issues placing new demands on information. Globalization is one such issue. Today, not only are many materials being sourced from overseas, but entire products or components thereof are being manufactured overseas, but to American designs and specifications. The need for information relative to equivalency of U.S. versus foreign materials test methods, delivery schedules, etc., is obvious. Some relevant information sources were included in Section 2. Others may be located through the International Forum on Globalization[6] and other Websites.[7,8]

A second factor is the growing use of so-called e-commerce: doing business almost entirely electronically rather than via printed documents or interpersonal information exchange. The extent of the use of e-commerce varies between fields, but current estimates are from 20–50% of all business conducted in nearly all fields and growing rapidly. This circumstance does not alter the kind or amount of material information needed but puts new demands on its currency and places enhanced emphasis on information already in electronic form.[9–11]

Finally, another current trend is that of "just-in-time" logistics management.[12–15] By this term is meant that the materials purchaser chooses not to buy and store materials at his plant until needed by manufacturing operations but rather contracts with one or more vendors to deliver a needed material on a continuing basis, obviating on-site storage and its associated costs, and feeding that material immediately upon receipt into the manufacturing operation. Again the associated information is not different in kind from that needed for traditional manufacturing, but new emphasis is placed on currency and intimate mutual understanding of information exchanged between vendor and manufacturer.

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PART 4

TESTING AND INSPECTION

Handbook of Materials Selection. Edited by Myer Kutz

CHAPTER 19

TESTING OF METALLIC MATERIALS

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One of the daunting challenges that face a designer when making a material selection is knowing what properties are the most critical for a given application. Assuming that the appropriate properties can be chosen, the designer next needs to be able to find the properties for each metallic material of interest (a challenge in itself) and then interpret the values relative to the design requirements. This requires that the designer have rudimentary knowledge of both the properties and test methods as well as an understanding of how to interpret the properties in the design context.

The purpose of this chapter is to provide some of the basic background for how metallic materials are mechanically tested. The goal is not to provide enormous detail for each different type of property; other sources, for instance Ref. 1, can provide this. Rather, the intent is to provide sufficient basic information so that the reader understands what the purpose of the mechanical test is, what mechanical property typically results, and what the corresponding design implication is.

The complexity of metallic materials testing and the specific technical language required can be confounding, especially to engineers not intimately familiar with the field. Those of us in the field also can confuse each other as new methods, terminologies, and approaches develop and are adopted. Luckily, there exists an organization whose mission in part is to minimize this type of misunderstanding.

Handbook of Materials Selection. Edited by Myer Kutz

The American Society for Testing and Materials (ASTM) is one of the world's largest standards development organizations with over 34,000 members responsible for more than 10,000 standards. The breadth of these standards is enormous (74 volumes), covering not only test/evaluation procedures but also standardization approaches for designating metals, paints, plastics, and the like. Originally developed in 1898, this independent, not-for-profit organization serves as a forum for producers, users, consumers, and others in developing voluntary, consensus standards. In practice, a hierarchical committee organization develops the standards initially with a group of experts providing a draft standard. This draft is subsequently balloted by ASTM and does not become a standard until all concerns raised during the voting process have been fully addressed.

The test-related standards that ASTM publishes help to ensure that mechanical properties generated in one laboratory are consistent with those generated in another laboratory. This consistency is critical from a design point of view since specific property values are typically extracted from numerous sources, and knowing that consistent methods were used in generating these properties is subsequently crucial. The majority of the ASTM standards related to metals test methods and discussed in this chapter are contained within Ref. 2.

Knowing where to look for the standard does not necessarily imply that it is easy to know which property is the most germane for a given design. For instance, under the classification "fracture testing," there are 16 separate standards in Ref. 2, all very different and evaluating disparate quantities such as crack-tip opening displacement, plane-strain fracture toughness, surface crack toughness, dynamic tear properties, and the like. The fundamental characteristics of the design scenario involved and the candidate metallic materials considered will dictate which fracture property is suitable. In a case such as this where many different test methods are available, selection of the appropriate test standard is complex and beyond the scope of this elementary treatise.

Finally, ASTM also provides other resources to ensure that the properties required are available. In particular, the *Directory of Testing Labs*[3] provides a state-by-state listing of laboratories that are available to perform materials testing. The laboratories are identified by the type of test performed, with an index cross-referenced to specific test methods. Moreover, a brief synopsis of the lab, including scope of testing, facilities, and staff, is also provided to allow differentiating between the different available choices.

1 MECHANICAL TEST LABORATORY

During mechanical testing, a component or material is loaded with either displacement or force, causing a deformation and associated mechanical response to occur. The loading occurs at a design relevant rate (impact, static, or cyclic), and the test is typically performed in the most suitable environment for the end use of the component. The two key components in this process that make up the primary constituents in a mechanical test lab (Fig. 1) are the test machine required to apply the specified loading as well as the sensors and instrumentation used to measure the behavior of the test article.

1.1 Test Machines

The test machine, or loading frame, is used to apply stress (or strain) resulting in axial tension or compression, bending, shear, torsion, or pressure. Although

Fig. 1 Typical material test lab containing numerous servohydraulic test frames. (Courtesy of Fracture Technology Associates)

a variety of different loading systems are available, the most common is the universal testing machine, which allows all of these different types of loading types by use of different test fixtures and load train components. Load is applied through a hydraulic piston and cylinder (servohydraulic machine) or with precision machine screws (screw machine) driven by an electric motor and gear box system. Although screw machines have lower load capacities than servohydraulic systems (generally 125 kips or less), their displacement stability, accuracy, and precision is better than typically observed with servohydraulic systems. Nevertheless the advantage of the more common servohydraulic systems is the rapid rate of response necessary for many types of tests (e.g., any type of cyclic testing, including fatigue and fracture applications).

For the case of servohydraulic machines, the actuators are typically double-ended allowing tension and compression of the load train. The displacement rate of these machines range from nearly static to approximately 350–1000 in./s. The actual highest rate possible for a given machine/specimen combination is difficult to assess since it depends upon the actuator size, flow rating of the servovalve, as well as the supply pump, plumbing configuration, and control system parameters. Servohydraulic test machines range in size but are typically available from 1 kip to 1000 kips or more.

The control of screw and servohydraulic machines is accomplished through dedicated microprocessor-based electronics. Controllers generally fall into two categories: the older generation (but stable and cost effective) analog controller and the newer digital controller (Fig. 2). In the strictest sense, an analog controller needs a signal source to drive it, typically either a built-in function generator or an external computer for complex waveforms or more stringent control requirements. Whereas machine control in this case is with analog electronics, in the fully digital system closed-loop control is achieved with a computer running software designed to perform the test of interest. The digital systems are typically provided with a suite of programs designed to perform a wide variety of standard test protocols.

A test machine is incomplete without a range of grips and fixtures suitable for inserting a test specimen into the load train of the frame. Although the

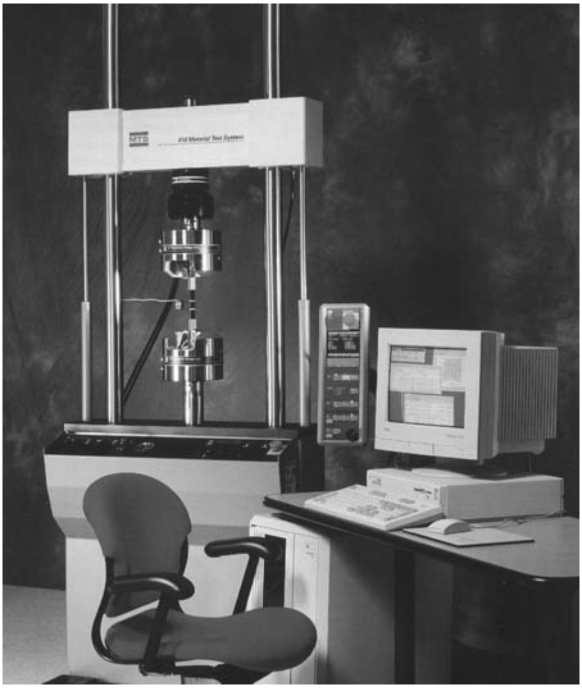

Fig. 2 Servohydraulic test frame and digital controller. (Courtesy of MTS Corporation)

majority of the ASTM test standards address fixtures and grips in some manner, inevitably the hardware available in a given laboratory is diverse and varied, consisting of both standard and hybrid fixtures whose functionality has been proven through repeated use. Versatile pneumatically or hydraulically actuated mechanical clamp grips constitute one of the more popular types available.

1.2 Sensors and Instrumentation

The sensors and instrumentation in a lab are designed to measure mechanical behavior. Perhaps the most commonly used sensor is the load measuring device, also called a load cell. Load cells are placed in series with the specimen in the loading train and can come in a wide variety of sizes and configurations. Normally, threaded couplings are available on either side of the load cell to attach fixtures. Inside the load cell is an instrumented transducer, typically using strain gauge technology to relate applied load to a resistance change suitable for sensing.

Although numerous nontraditional methods and systems exist for measuring displacement or strain,[4,5] the most common method is based on a sensor that provides output based upon strain gauge technology. An example of this is the highly reliable modern material test extensometer (Fig. 3). This device, fastened to the specimen by mechanical clamping, is used to accurately measure displacement between a pair of short knife edges affixed to the gauge length of the specimen. Another type of displacement gauge is the clip gauge, which is basically a spring-loaded extensometer with longer arms. These devices are

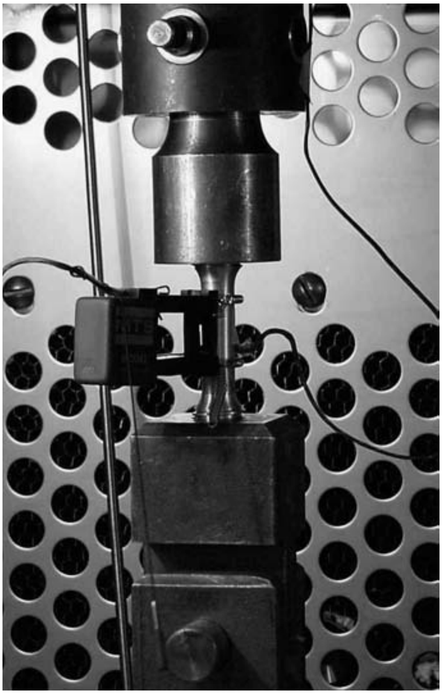

Fig. 3 Extensometer (displacement measurement device) mounted on a tensile specimen.

extremely accurate and robust, but they do have environmental limitations to some extent due to their reliance on strain gauge technology.

Sometimes it is also useful to mount strain gauges directly on the surface of the test specimen to infer localized behavior. Furthermore, linear variable differential transformers (LVDTs) and capacitive coupling-based displacement devices are commercially available for use in the most aggressive environments. In addition, noncontact optical systems (laser or charge coupled device, CCD, camera based) can also be used to obtain displacement measurements. Recent improvements in computer processor speed, CCD camera size, as well as more affordable random-access memory (RAM) and disk storage makes these vision-based technology methods more desirable now more than ever before. The preponderance of optical systems and lack of any standardization in the core technology has resulted in recent ASTM standards activity and a draft standard that defines the relevant terminology.

Data acquisition tools have also benefited greatly from the recent advances in personal computers. Newer data acquisition systems tend to be more flexible than the older, device-dependent applications. The most popular platform for application building currently appears to be the LabVIEW (National Instruments, Austin, TX) programming environment. The LabVIEW platform provides a graphical, icon-based environment to build and modify applications that are more device/board independent and well suited to typical complex process, and data acquisition requirements.

Some of the most complex tests performed in a mechanical test laboratory are fatigue and fracture evaluations of metallic components and materials. These tests usually require highly specialized, custom software to control the test machine, take the data, and analyze the results. It is always difficult to know *a priori,* without extensive experience performing these tests, what data acquisition parameters are the most critical. For this reason a recent guide, ASTM E1942, has been developed that assists a user to understand the limitations of instrumentation and data acquisition systems, especially as related to cyclic fatigue and fracture mechanics testing. Furthermore, this standard is also useful for the segment of the test community not familiar with fatigue and fracture tests, since it provides a practical guide for understanding data acquisition performance.

2 TENSILE AND COMPRESSIVE PROPERTY TESTING

The tension test, defined for metallic materials in ASTM E8, is perhaps one of the simplest and most common tests of mechanical behavior. The test is conducted by gripping each end of a reduced section specimen and slowing pulling it until catastrophic failure occurs. Several sample specimen geometries (round and flat, threaded and smooth shank), conventionally described as ‘‘dogbone’’ specimens by virtue of their reduced section geometry, are shown in Fig. 4. Moreover, tension test specimens are sometimes notched in the center of the gauge length as described in ASTM E338 and E602. These tests are useful for determining how the given metallic material behaves in the presence of a stress concentration. Furthermore, results from these tests have also been related to fracture mechanics parameters for a given material.

In a tensile (or compressive) test, the applied load and displacement (or strain) in the gauge section is used to develop a plot of the stress–strain behavior using the specimen geometry and calibration constants of the transducers employed during the test. One challenge during tensile testing is measuring elongation to

Fig. 4 Several sample specimen geometries for tensile testing in accordance with ASTM E8. (Courtesy of the Instron Corporation)

high strain levels while still having sufficient sensitivity in the elastic region. It is therefore not uncommon to utilize two strain transducers as shown in Fig. 5. Special high-gain strain gauges or transducers are typically required to provide sufficient sensitivity and resolution in the elastic region to derive a Young's modulus value. This is contrasted with conventional extensometry, which is usually used to measure higher levels of strain, even 20% or higher. Alternatively, two extensometers, one measuring behavior over a small range while the other configured for a large range, can be mounted on a single specimen. One advantage with this approach is that strain differences as a consequence of different gauge lengths, due to issues such as strain localization, is greatly reduced since each extensometer can have the same basic gauge length but be calibrated to different ranges.

During tensile testing, the ultimate strength is the largest applied stress measured in the specimen section. The proportional limit, often called the yield strength, indicates the onset of plasticity where the assumption of linear stress–strain behavior is violated. Since there are many definitions of proportional limit (e.g., the conventional 0.2% strain offset method or the API approach[6] using the stress corresponding to a specific strain level), it is imperative that the physical meaning of the limit be clearly defined when presenting the results. Moreover, post-test measurements made on the gauge length and fracture surface of the specimen are used to quantify the ductility of the specimen. Ductility is the ability of a material to deform plastically without failure occurring. Ductility parameters include percent elongation and percent reduction of area.

The mode of failure can also sometimes influence the strain results obtained. For instance, if the metallic material tested tends to exhibit highly localized plastic deformation, the region of confined necking results in either higher or lower effective strain if the gauge length is short or long, respectively. On the other hand, if deformation is equally distributed along the gauge section, the

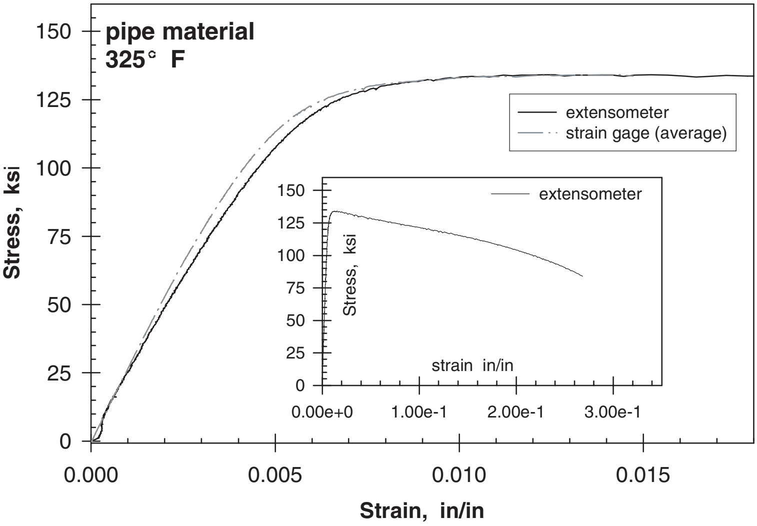

Fig. 5 Example stress–strain data from a tensile test from a pipe material tested at 325°F.

strain data exhibits no gauge length dependence. Obviously, the potential exists for misinterpretation when comparing stress–strain behavior from one sized specimen to another. Therefore, it is important to ensure that specimen size is constant when performing a relative comparison of properties from one condition to another or between metallic materials.

Compression testing is typically performed on short cylindrical specimens (ASTM E9). Care must be taken during testing to minimize the influence of both buckling and barreling, two modes of behavior that can corrupt compression test results. Buckling is prevented by minimizing the ratio of specimen length to diameter (typically in the range of 1–2). Load train alignment is also a critical feature of these tests. Alignment is often practically achieved by loading the specimen through a spherical joint so that slight, nearly unmeasurable misalignment will not cause buckling. Barreling results from the friction between the end faces of the specimen and the loading platens. Friction is minimized by using lubricant and machining concentric rings on the end of the specimen to trap the lubricant and keep it from squeezing out.

3 CREEP AND STRESS RELAXATION TESTING

Creep and stress relaxation properties are critically important for high-temperature applications. Without knowledge of these properties, both air- and land-based jet turbines, internal combustion engines, and even some electronic equipment could not be adequately designed. Furthermore, there is always increasing pressure to develop new metallic materials with better performance at high temperature so that operating temperature in these applications can be increased to yield higher efficiencies.

Creep deformation is examined by applying either a constant load or a constant true stress to a material held at a specified temperature. When the material is first loaded, a small permanent loading strain occurs and the creep strain rate gradually decreases. This portion of the creep strain versus time plot is called primary creep (stage I). This is subsequently followed by secondary (or also steady-state or stage II) creep with the highest rates observed in the final tertiary creep (stage III) immediately before failure. These three regions of the creep curve are shown schematically in Fig. 6. Whereas the test methods are described in detail in ASTM E139, the primary components of the test include a static load frame, furnace, and high-temperature extensometry.

The test specimens are similar to those used in a quasi-static tensile test, although the actual geometry used is often controlled by the metallic material form available. Static dead-weight machines are typically employed for this type of test, as shown in Fig. 7. The frames usually have a balance beam to magnify the load applied to the specimen by a factor of between 2 and 25. All of the pull-rod and coupling fixtures employed are manufactured from high-temperature alloys impervious to the environment used during the test. Tubular, electric resistance furnaces are usually used with the number of zones typically dependent upon the size of the specimen. Special attention must be made to ensure minimal thermal gradients and uniform temperatures applied to the specimen.

The measurement of gauge length deformation is typically critical during a creep experiment. If temperatures are sufficiently low (<400°F), conventional

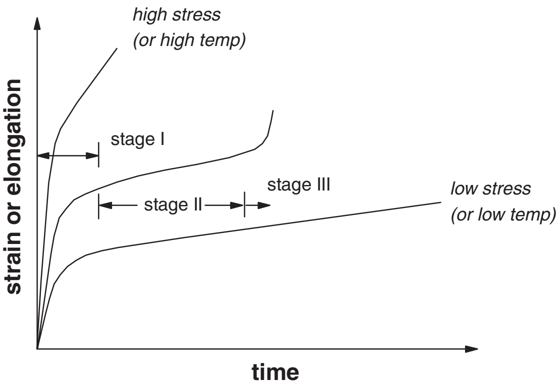

Fig. 6 Schematic of the different regions of a classic creep curve.

Fig. 7 Loading frame to apply static (or extremely slow cyclic) load conditions. (Courtesy of the Instron Corporation)

extensometry as used in tensile tests can be used on the specimen. However, higher temperatures are usually involved for most metals, which necessitates using LVDT, capacitive, or other types of displacement measurement transducers. The most common transducer is the LVDT coupled with linkages that reach up through the furnace to attach to the specimen. Special care must subsequently be taken to ensure that the displacements measured actually correspond to gauge length deformation and are not biased by linkage or transducer effects.

Stress relaxation testing (ASTM E328) is similar in many ways to creep testing except that the applied boundary condition is a constant displacement (as opposed to a constant load). This type of test is more suitable for metallic materials that have significant constraint and hence exhibit little strain increase as a function of time. One example of this is a high-temperature bolted joint where a displacement boundary condition is first applied and then the stress relaxes with time. Testing times include both long-term (months or more) at the service temperature and accelerated tests (days) at more aggressive thermal conditions.

Creep and stress relaxation testing can also be performed on specimens with a more complex stress state than simple tension. Examples include compression testing (typically using a carefully machined cylindrical specimen similar to that described earlier for quasi-static compression testing), bend testing (e.g., with a cantilever beam specimen), or even torsion testing. Testing has also been performed on tubular specimens that are subject to complex, multiaxial stress states.

4 HARDNESS AND IMPACT TESTING

This section addresses two mechanical test methods that are used to indirectly determine the tensile and toughness properties of a metallic material. As with any indirect test method, there are well-known limitations to these techniques, but they are clearly offset by the reduced cost and increased efficiency of the indirect methods. The test methods, as well as their limitations, will be explained in more detail in this section.

Hardness refers to a material's ability to resist deformation when loaded by an indenter. The indenter can differ in geometry; for instance, it can be conical (the Rockwell test), spherical (Brinell), or pyramidal (Vickers and Knoop). For metallic materials, hardness is directly proportional to the yield stress of the material. Nevertheless, conversions are readily available to relate hardness to ultimate tensile strength (most commonly for steel although other conversions for different metallic materials do exist). Hardness testing (ASTM E6, E10, E18, E92, E1842, and others) is industrially attractive since the testing is rapid (<20 s), low cost, nondestructive, and generally does not require a machined specimen. As a consequence of these issues, hardness is often used as an industrial screening tool to ensure metallic material processing control. An ASTM standard (E140) exists to convert between the different superficial hardness and conventional hardness scales.

The majority of hardness testers (e.g., those shown in Fig. 8) utilize deadweight, springs, or (infrequently) closed-loop computerized systems. Although many testers provide output on an analog meter, bench-top units with digital readouts (sometimes with an interface for a PC) are generally more popular today. When the component being hardness tested is too large to practically


(a) (b)

Fig. 8 Hardness testers including (a) a benchtop and (b) portable unit. (Courtesy of the Instron Corporation)

position onto the anvil of the tester, portable test units are available to locally measure hardness. A typical portable hardness measurer is shown in Fig. 8*b*. Regardless of the type of hardness tester used, care must be taken to ensure that the system is in proper working order by using commercially available calibration blocks.

Although hardness testing is relatively simple, accurate measurements depend greatly on careful attention to detail. The anvil should minimize the contact area with the component. Surface finish can sometimes be of paramount concern; for 100–150 kgf, a finish ground surface is sufficient whereas for a 15-kgf load, a polished or lapped surface usually is required. Surfaces that are ridged due to machining or that exhibit loose, flaking scale can yield incorrect hardness results. The test surface should be perpendicular to the indenter since even slight ($<5°$) angular deviation can cause hardness to shift by several points. Indenter size should be chosen with the underlying grain structure of the metallic material in mind. In general, the indenter should be larger than the grain size if a macroscopic measurement of hardness is desired. Finally, metallic material homogeneity is also important since the indenter effectively samples a region approximately 10 times deeper than the depth of the indenter penetration.

Instrumented indentation testing is a more recent method developed to derive more extensive data from effectively a hardness test. During this type of testing, the instantaneous load–displacement data is recorded continuously. The measured data can be related to such things as elastic modulus, yield strength, and strain hardening exponent. Highly empirical punch test methods are also continually evolving to relate local deformation behavior to properties such as fracture toughness.

Since some percentage of loading is inevitably dynamic and components often have to be designed to survive high loading rates typical during an accident, dynamic fracture testing can ensure structural integrity under this type of loading. During impact testing, a pendulum machine (example shown in Fig. 9) with a striker is typically used to impart high energy to a notched sample. The notch toughness of the specimen, typically a flat bar with a carefully machined notch of various geometries, is measured in terms of the absorbed impact energy that causes fracture in the specimen. Some other parameters that are commonly examined include fracture appearance and the deformation noted on the surface of the specimen. The independent variable controlled during testing is most often temperature. A typical energy–temperature curve for A36 steel is shown in Fig. 10 contrasting the typical difference between slow bend tests and rapid impact results.

The Charpy and Izod impact tests both utilize pendulum loading albeit with specimens of slightly different geometry, especially as related to the specimen notch dimensions. The primary difference between the two tests though is that the Charpy V-notch (CVN test as defined in ASTM E23) specimen is three-point loaded whereas the Izod specimen is cantilever loaded.* Of the two methods, the CVN test remains the most popular method available, and manufacturing

Fig. 9 Example of a pendulum impact machine. (Courtesy of the Instron Corporation)

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*Both machines were developed in the beginning of the twentieth century although Charpy was a Frenchman and Izod was an Englishman.

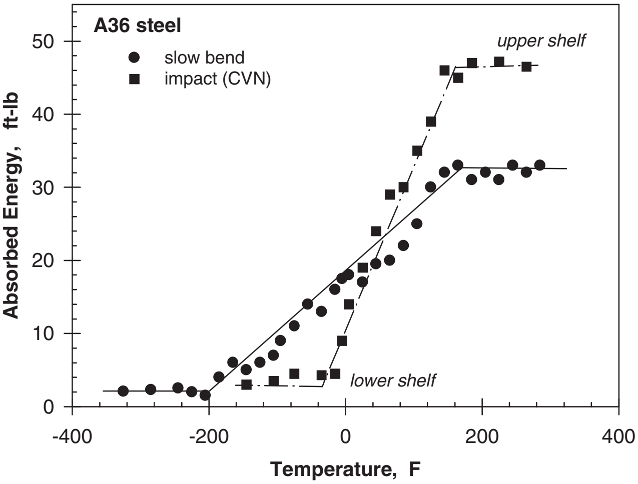

Fig. 10 Impact test results illustrating CVN energy versus temperature curves for A36 steel.

lot release requirements using CVN data are actually written into many metallic material specifications. The CVN test is rapid and low cost; the most expensive part is machining specimens although commercial machining templates to ease fabrication are readily available.

Two other impact tests include the drop-weight (ASTM E208) and the drop-weight tear test (DWTT as per E436). During drop-weight tests, a series of experiments are performed where different specimens at a range of temperatures are impacted with a guided, free-falling weight dropped from a fixed height. The key finding is the nil-ductility transition temperature (NDTT), which is the maximum temperature where fracture was observed. The DWTT test is similar to the drop-weight test although the specimen used is slightly larger and the test fixturing is very different. The fracture surfaces are evaluated and the percentage of shear quantified. The temperature at which 50% shear occurs is loosely defined as the ductile-to-brittle transition temperature.

CVN testing can also be performed with a fatigue precracked specimen to eliminate the notch acuity and depth restrictions inherent to the standard CVN specimen geometry. Considerable effort has also been recently expended developing instrumented impact testers to record more data. Nevertheless, the primary shortfall with most of the impact test methods is (a) the relatively small specimen size (relative to component size) and (b) the high degree of empiricism used to relate the results to metallic material properties. These disadvantages are offset by the simplicity and low cost of the test, particularly for production environments where the test can be used effectively as a quality control tool.

5 FRACTURE TOUGHNESS TESTING

The foregoing discussion introduced the concept of notch toughness, defined loosely as the energy required to initiate a running fracture from a machined notch. This is an important design parameter since nearly all structures contain notchlike stress concentrations. However, in the strictest sense according to the science of fracture mechanics (the study of cracks), fracture is broadly classified as either brittle [governed by linear elastic fracture mechanics concepts (LEFM)] or ductile [elastic plastic fracture mechanics (EPFM)]. Fracture toughness is the ability of a material to withstand unstable crack propagation (e.g., fracture) assuming of course the presence of a crack. Fracture mechanics provides the theoretical underpinning that relates the following quantities:

- Magnitude and range of the remote applied stress
- Geometry of the component
- Morphology (size and shape) of the crack

to what is loosely termed the "crack driving force," or stress–intensity factor K. Detailed stress analyses of cracks have shown that the magnitude of the local crack-tip stress field is proportional to this K parameter. Although K applies for LEFM, in the case of EPFM there exists another similitude parameter that is termed J.

Fracture toughness testing requires closed-loop, computer-controlled servohydraulic test equipment and a suite of accurate displacement extensometers. Virtually all tests are computer controlled due to the complexity of the test and the requirements for recorded data. As a minimum, most fracture tests require measuring the applied load and some displacement measurement across the crack. Most tests are analyzed by custom software developed specifically for the particular test. The most common fracture toughness test is the plane-strain fracture toughness measurement, which yields the K_{Ic} parameter as described in detail in ASTM E399. For an ASTM valid K_{Ic} value, the loading must be such that there is no significant plastic deformation (specifically, the plastic zone at the tip of the crack is less than 2% of the thickness and total crack length). The plane-strain requirement implies that the fracture toughness specimens tend to be quite thick, most are commonly greater than 0.5 in.

Although E399 allows a variety of specimens, the most commonly used is the compact-tension specimen, a pin-loaded, edge-notched geometry. The specimen is first precracked under fatigue loading to ensure that the crack tip is sufficiently sharp for the fracture test. The specimen is then loaded to failure by a constant displacement ramp applied between the pins. During the test, the load and crack mouth opening displacement (CMOD) is measured. Two sample load–displacement plots are shown in Fig. 11. In Fig. 11*a*, the behavior is more brittle with catastrophic failure at peak load. Conversely, in Fig. 11*b* the trend is more suggestive of a ductile and somewhat more stable material with slow crack advance gradually relieving load as the test progresses past the point of maximum load.

Determining a K_{Ic} toughness from the load–CMOD data requires fitting the initial linear portion of the data and plotting a 5% secant offset to that linear region. The intersection between the measured load–CMOD data and this secant line defines the critical load. This graphical construction is evident in Fig. 11*a*. However there are stringent limits for qualifying the conditional K_q toughness as an ASTM-valid K_{Ic}. Most importantly, the crack length and thickness are required to be sufficiently long to satisfy the small-scale yielding criteria de-

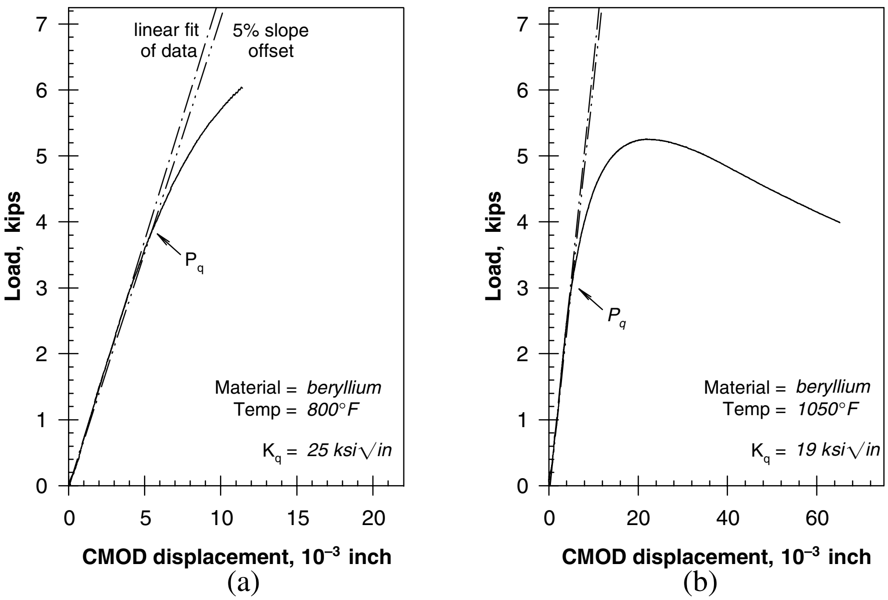

Fig. 11 Two load–displacement plots from K_{Ic} fracture toughness tests conducted on beryllium at elevated temperature.

scribed earlier. Furthermore, nonlinearity is minimized by requiring that the critical load (corresponding to K_q and denoted by P_q) must be no less than 90% of the maximum measured load. The two sets of data indicated in Fig. 11 are clearly invalid since the conditional P_q load is too low relative to the maximum measured load.

Whereas K_{Ic} applies for thicker, more constrained sections where catastrophic fracture occurs rapidly, a different K_C fracture test (ASTM E561, also termed a *K–R* curve) applies for thinner structure where slow stable crack extension precedes unstable fracture. The test specimen geometry typically used is a wide, center cracked panel precracked at low loads to generate a sharp fatigue crack suitable for fracture. The specimen is monotonically displaced similar to a K_{Ic} specimen while recording load, CMOD, and often some parameter such as electric potential drop that can be used to instantaneously provide crack length measurement.

The resistance curve, also called an *R* curve, is then generated by plotting crack driving force *K* against crack extension, termed Δa. Crack extension is defined as the sum of the physical crack length change, usually derived from either compliance (stiffness) or some other nonvisual crack length indicator, plus the half plastic zone size. Since the stress state is typically something more like plane stress, the plastic zones as observed on the surface of the specimen tend to be quite large. An example R curve is shown in Fig. 12. The K_C value is defined as the *K* magnitude at the tangent intersection between the *R* curve and lines of constant load plotted in *K* versus crack length (or crack length extension) space. This load represents the maximum level that can be achieved before the crack extends (often unstably) and structural integrity is lost. A careful exami-

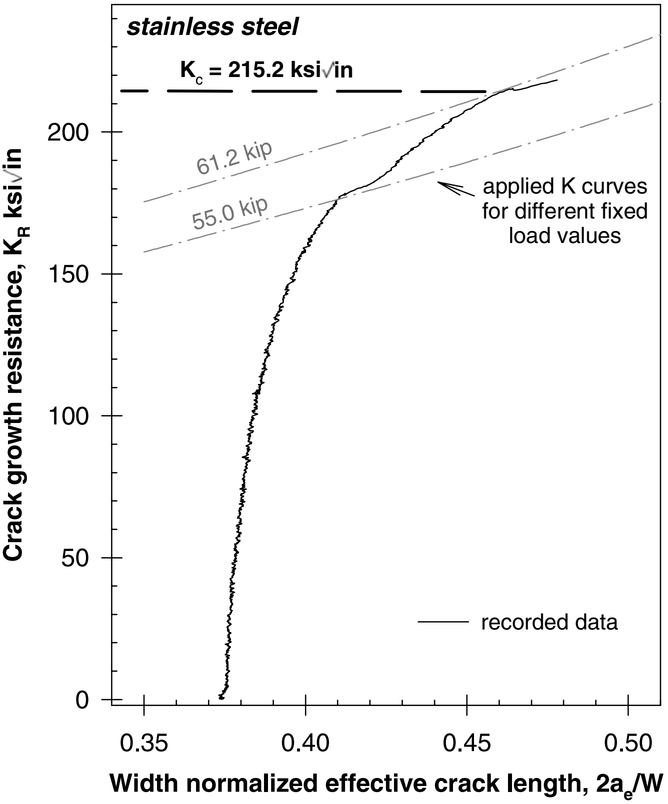

Fig. 12 Example *R* curve from a K_C fracture toughness test performed on stainless steel.

nation of the data in Fig. 12 clearly illustrates that some crack growth was apparent in this metallic material since catastrophic failure did not occur at the point of instability.

Fracture toughness testing in the elastic-plastic regime is time consuming, not trivial, and requires advanced instrumentation to yield excellent results.[7] After conducting and analyzing the test, the goal is to generate an *R* curve, similar to that described earlier for the K_C testing, as shown in Fig. 13. However, instead of *K* on the abscissa, the *J* parameter is included. The *J* parameter is calculated by including an elastic portion related to *K* combined with a nonlinear portion related to the area under the load–displacement curve (with respect to load-line displacement). The plane-strain, elastic plastic J_{Ic} parameter measures the onset of instability, in this case corresponding to the point where the crack has advanced a certain amount (after blunting) on the *R* curve (note this instability criteria is very different than that described for K_C). It is often the case in this type of test that slow, stable cracking will continue as the specimen is displaced and the load gradually drops to a small fraction of the peak obtained.

The J_{Ic} test procedure is usually performed using compact-tension specimens, although three-point bend specimens are also possible provided care is taken to ensure that accurate instrumentation is available to measure load-line displace-

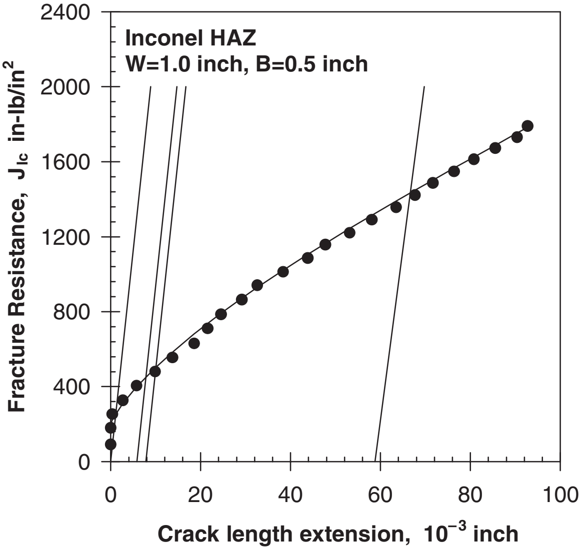

Fig. 13 Example of an *R* curve from a J_{Ic} fracture toughness test performed on a high-strength weld.

ment. The primary challenge with J_{Ic} testing is the high accuracy required from measurements of the load-line displacement. High-resolution measurement is required so as to infer the extremely small amount of crack tearing necessary for the J_{Ic} fracture toughness definition. This high accuracy is practically achieved through higher accuracy (minimum of 16 bit), low noise data acquisition boards combined with extreme care and control over the critical test details, especially as related to loading pin interfaces and clip gauge attachment.

In editions of the ASTM test standards prior to 1998, the test method for the elastic-plastic J_{Ic} parameter has always been described in ASTM E813. However, the E08 committee of ASTM has recently published a combined approach under the designation E1820. This combined approach outlines a procedure to follow that can result in any one of the primary toughness parameters, namely K_{Ic}, *K*-, and *J*-resistance curves. Although these three parameters constitute the most common fracture test standards, other ASTM methods are also available:

- Crack-tip opening displacement (CTOD) fracture toughness (E1290)
- Plane-strain crack arrest fracture toughness (E1221)
- Fracture testing with surface-crack tension specimens (E740)
- Chevron notch fracture toughness of metallic materials (E1304)

However, these methods are typically not as wide-ranging nor as commonly encountered in design as the previously noted fracture toughness test methods.

6 FATIGUE TESTING

The previous section described basic fracture mechanics concepts as well as the test methods used to quantify fracture behavior given a preexisting crack. In this section the process of crack nucleation (e.g., crack initiation) and stable propagation (e.g., crack growth) under cyclic loading is addressed. Understanding crack propagation behavior is critically important since some structures, most notably the current fleet of commercial airliners, adopt a damage tolerance design philosophy whereby cracks are assumed in the structure, and a safe structural life requires detecting and managing these cracks. Quantifying crack nucleation is also important since much of a crack's cyclic life is spent initiating or growing when it is small in size.

The test specimens used for fatigue crack initiation testing are similar geometrically to tensile specimens. The two basic test methods for initiation testing are ASTM E606 and E466 for strain- and stress-controlled testing, respectively. With both of these methods, the goal is to cycle a specimen until failure occurs. Strain control is typically used if (a) the loading is with a constant displacement or (b) the focus is on the short life regime (estimated to be less than 10^3 to 10^5 cycles although this is fairly controversial) where applied stress levels often approach the yield strength of the metallic material. Conversely load control testing is performed for the mid- to high-cycle life regime. Stress (effectively load) control testing is relatively straightforward since no displacement monitoring or measurement is required. The specimen can be gripped, cycling can commence, and continue unmonitored (provided the servocontrol loop does not require any attention) until failure occurs.

One of the most critical details when performing this type of fatigue testing is controlling bending in the specimen. Poor control of bending typically increases the apparent scatter in the fatigue data generated. Commercial alignment fixtures are available to mitigate bending. These fixtures utilize a strain gauged specimen and a fixture that allows minute adjustments of the loading train. However, these fixtures are not always available, which means less high tech and pragmatic methods are often required to minimize the effect.

Although guidelines on how to measure and verify specimen alignment are provided in ASTM E1012, minimizing bending practically requires control of three issues. First, the specimen must be carefully machined with excellent dimensional control, parallelism, and axisymmetry. Second, the fixtures used to connect the specimen to the loading train must not introduce any asymmetry. Threaded specimen ends, although commonly employed during fatigue testing, should be avoided as the tolerances required to engage the thread make accurate alignment problematic (however, it must be stated that it is not always practical or possible to avoid threaded ends). If the loading cycle is tensile only (e.g., a positive R ratio where R is the ratio of minimum to maximum load), a spherical joint, or more commonly known as a U-joint, in the loading train can minimize bending. Third, the fixtures in the load train should be designed well enough to grip repeatably in the same manner so that once a load train has been carefully aligned with an instrumented specimen it does not vary.

Some typical stress–life (S–N) data are shown in Fig. 14 for two materials, an aluminum and titanium alloy. S–N data are usually generated at a fixed R ratio with the results represented as a plot with stress amplitude or maximum stress on the abscissa and cyclic life on the ordinate. Data are sometimes generated on specimens that have a groove or hole to simulate stress concentrations typically found in structure. Furthermore, it is often of practical interest to know

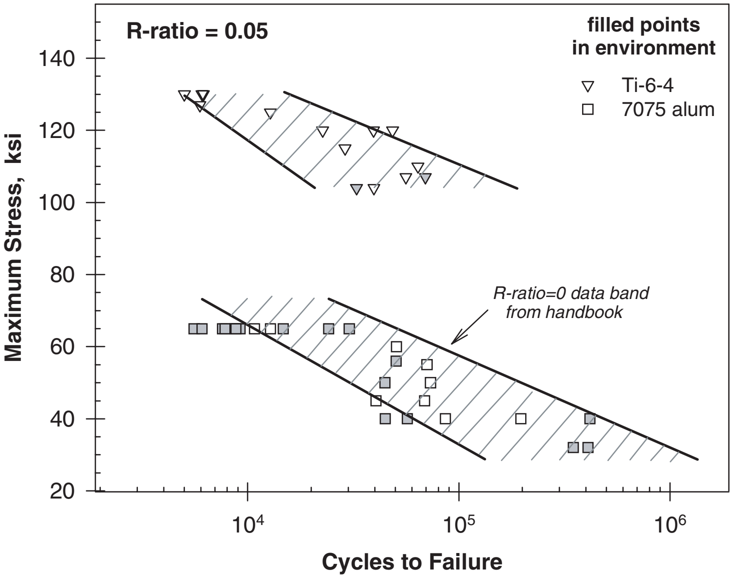

Fig. 14 Conventional *S–N* fatigue test data presented for example titanium and aluminum alloy materials.

the endurance limit of a metallic material; in theory the endurance limit is defined as the applied stress level that corresponds to infinite life, in practical terms infinite life is approximated in the laboratory as a million cycles. The usual approach[8] is to test 30 specimens using an iterative, staircase method to converge on the stress level corresponding to the endurance limit. The nominal stress step chosen is approximately 2% of the ultimate tensile strength of the metallic material. Although this is a test-intensive methodology, the technique is able to clearly define the endurance limit to the accuracy given by the stress step.

The specimens used for fatigue crack propagation testing are similar to that described previously when discussing fracture. The most common geometries are the compact tension, C(T), and the middle cracked tension, M(T), specimens. However the pin-loaded C(T) is most suitable for positive R ratio (e.g., tension) since the cracked geometry is unstable in compression. Fatigue crack growth (FCG) testing is described in detail in one of the longest and most complex ASTM test standards, ASTM E647. The standard has grown in length as the understanding of the fatigue crack growth process has deepened. During FCG testing the goal is to determine crack growth rate as a function of stress intensity factor range, ΔK. A typical series of fatigue crack growth curves are shown in Fig. 15 for an aluminum alloy subject to a number of different stress ratios. The central regions of the crack growth curves can be observed to be relatively linear as expected in the moderate growth rate Paris regime.

One of the most critical aspects of fatigue crack growth testing is measurement of the crack length. The most common method is to use a traveling telemicroscope mounted on a vernier scale for accurate measurement. Recent advances in both crack length measurement and test automation[9,10] employ elas-

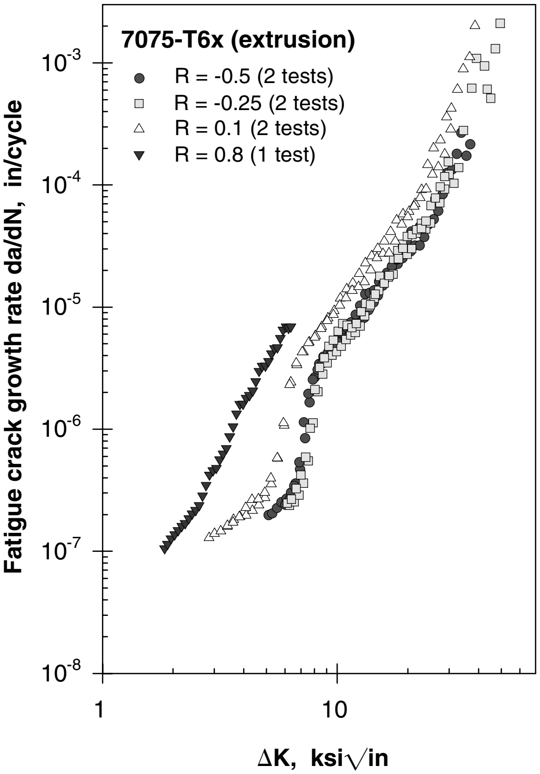

Fig. 15 Series of fatigue crack growth curves for aluminum at various *R* ratios.

tic compliance and electric potential drop methods to measure crack length. However, it is critical to note that these indirect, nonabsolute measurement techniques both require periodic visual crack length measurement for each test to ensure proper calibration. The quality of a laboratory's nonvisual crack length measurement technique and equipment is critical and often directly related to the quality of the FCG data generated.

The conventional method for performing crack growth testing is to record growth rate data with a fixed loading cycle as the crack grows. More efficient, newer methods are now available that critically depend upon nonvisual crack length measurement methods to control the applied loading. For instance, K gradient techniques can be used where the quantity $C = dK/K\ da$ is fixed at different levels for different portions of the test. This increases efficiency since (a) it allows the generation of multiple crack growth data sets from the same specimen and (b) data can be generated in short growth intervals resulting in much faster data generation than with constant amplitude loading. However, these advanced fatigue crack growth test techniques must be carefully applied by experienced testing professionals who fully understand the limitations of this accelerated approach and have the instrumentation available to accurately measure crack length nonvisually.

Most fatigue crack growth rate testing is performed at a fixed load ratio since this variable does not change for many structures. However, another method gaining wide popularity is generating FCG data from the highest rates down to near threshold (e.g., crack growth rate at a very low magnitude, a quantity especially useful for design purposes) using a constant $K_{\max}$, increasing $K_{\min}$ loading condition. Consequently when the test starts, the R ratio is low (0.1 or so) and as the test progresses R increases until approximately 0.9 or more in the near-threshold regime. Whereas conventional fatigue crack growth threshold testing may take weeks or months to complete, this method results in a highly accelerated test and can be performed in a matter of days. Again, care must be taken to ensure that the choice of the variable test control parameters does not affect the magnitudes or trends of the fatigue crack growth rate data obtained.

For design purposes, it is sometimes necessary to understand the potential impact of external variables on FCG behavior. For this case, constant ΔK testing can be employed to determine effects difficult and time consuming to detect if a complete crack growth curve were generated. As observed in the data in Fig. 16, several regions of nominally constant ΔK are sequentially applied with the external environmental variable perturbed for each segment. This allows a better assessment of the effect of the variable since a statistically relevant number of crack growth rates are available for each segment. Although this is a highly

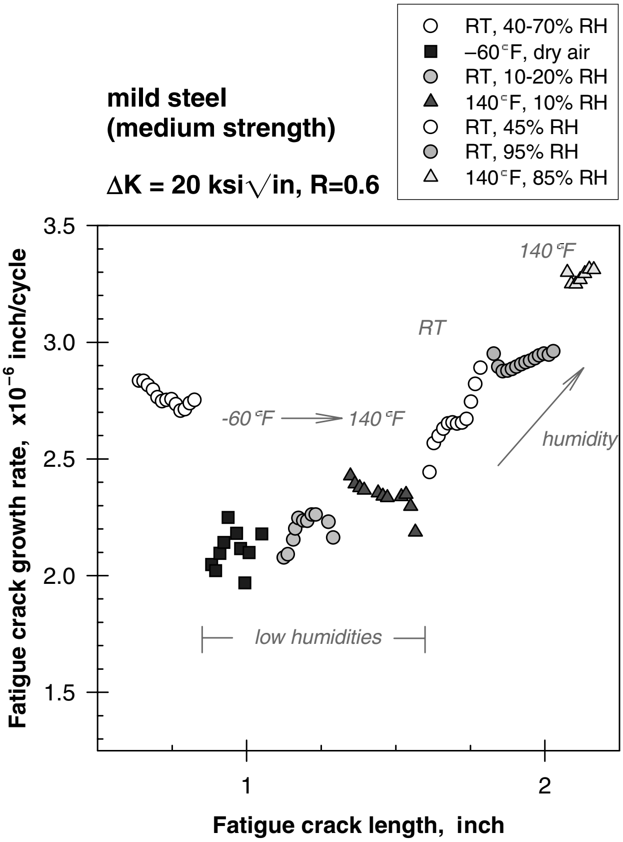

Fig. 16 Influence of environment on fatigue crack growth rate behavior using a constant ΔK testing approach.

efficient method to determine effects, the disadvantage is that the measurement strictly applies to only one constant ΔK level. Nevertheless, a similar approach can be used to indirectly determine, for instance, the magnitude of internal residual stresses in a component by examining small changes in fatigue crack growth rate.

The challenge in applying fatigue crack growth rate data is accounting for the variable amplitude loading environment typically applied to a component in service. Most of the analytical tools available are empirically based and require some testing under the variable amplitude, spectrum load condition. In these cases, it is not uncommon to perform a crack growth test using a repeated variable amplitude spectrum derived from either service recorded or design loading. The test specimens in these cases are typically more complex, reflecting some design detail such as a fastener hole or weld. Rather than generating crack growth rate data with these tests, the goal is typically to provide crack length as a function of spectrum passes (or cycle count). Although this type of test is widely performed in the aerospace and ground vehicle industries,[11] the myriad of approaches used has stifled standardization, and it is only now underway within ASTM to develop a standard practice applicable to this highly complex test.

7 OTHER MECHANICAL TESTING

There are a number of test methods and procedures that are related to the areas described earlier but, in the spirit of brevity, have been omitted. Nevertheless, several are sufficiently popular and warrant some mention, albeit brief.

Some highly specialized assessments other than tensile tests exist for determining the strength and ductility of a metallic material. One common application for the bending strength test is the use of flat metallic materials for spring applications (ASTM E855). The two primary properties that this test provides are the modulus of elasticity in bending and the bending yield strength (proportional limit). Moreover bend tests used to assess ductility (ASTM E190 and E290) do not provide quantitative results, but rather provide a deformed sample suitable for close visual inspection to determine basic metallic material behavior. The region of highest strain is typically inspected for possible cracking, rumples, or orange peeling as a quality control assessment suitable for the shop floor.

High strain rate testing is another area where significant effort has been expended to generate the constituent properties applicable in the high rate regime. For instance, the split Hopkinson pressure bar deforms a sample at a high strain rate (>100%) while applying uniaxial stress to the sample. Similar methods have been developed that also allow high rate torsional loading. These methods have been successfully applied to a wide range of metallic materials.

Finally, numerous test techniques are also available for testing adhesion, friction, and wear of surfaces. These are critical issues from a design viewpoint with new standardized methods being developed fairly regularly.

8 ENVIRONMENTAL CONSIDERATIONS

It is often necessary to perform mechanical testing under environmental conditions other than ambient air. For instance, fatigue crack growth testing is often performed in either dry, high humidity, or aqueous conditions. For aqueous con-

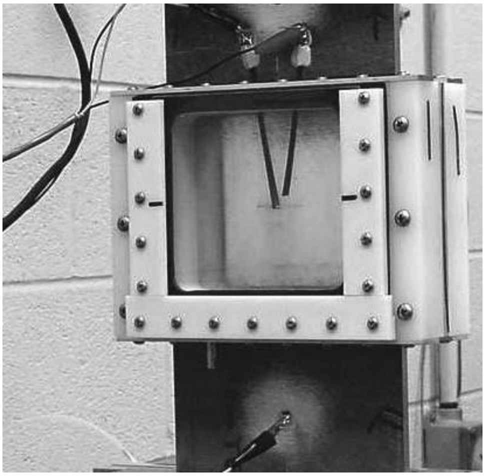

Fig. 17 Chamber around a flat panel M(T) specimen for suspending jet fuel or other aqueous environments while measuring fatigue crack growth rate behavior.

ditions, a setup as shown in Fig. 17 can be constructed to perform crack growth rate measurements. Sealing against a flat surface is relatively straightforward provided the materials used in the chamber are impervious to the environment contained.

Other strategies such as that shown in Fig. 18 can be used to seal the loading pins in a compact tension specimen. This technique has proven very suitable in the past for containing aqueous environments. Clear Teflon (PTFE) bags are commercially available that allow the setup to also be subjected to temperatures in the range of −400 to 300°F. Simpler methods using supermarket polypropylene bags can also be used to contain conditioned air by introducing either desiccant or a hydrated sponge for the dry and humid conditions, respectively. Although the engineer's inclination might be to build a more elaborate environmental chamber, a simple plastic bag with duct tape seals works sufficiently well

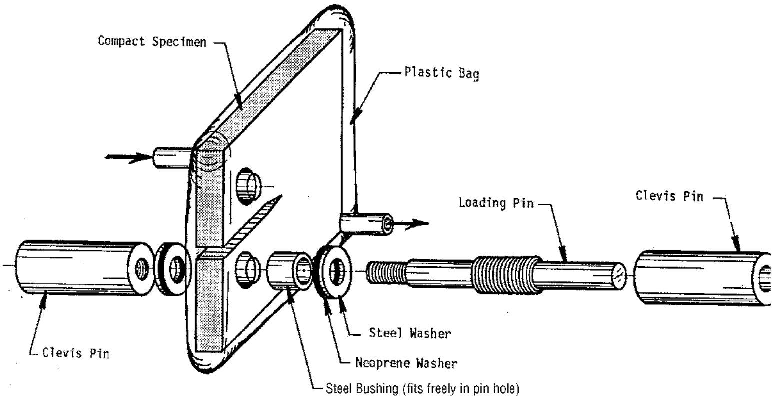

Fig. 18 Schematic of a compact tension specimen sealed with a plastic bag.

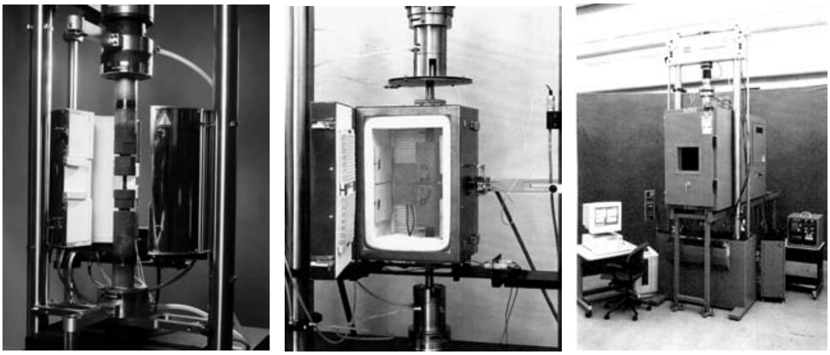

Fig. 19 Several examples of enclosures for both high- and low-temperature materials testing. (Photos courtesy of the Instron Corporation and MTS Corporation)

to contain dry [<5% relative humidity (RH)] and humid (>95% RH) environments.

Finally, it is sometimes not simple to superimpose two rate-dependent processes without special attention. For instance, care must be taken to ensure that the time-dependent effects that often occur in an environment are properly applied in the materials test. As an example, the deleterious effect of seawater is gradually lost during a fatigue crack growth test as the frequency exceeds 1 Hz.

If a geometry is somewhat unusual and a setup does not fit into existing furnaces, it is sometimes necessary to construct a custom furnace from insulating fiberboard so long as the temperature required is not too high. In this case, a circulating fan is included along with a heater element and inexpensive temperature controller. However, an inexpensive approach such as the one just described will not perform well given an aggressive environment, higher pressure applications or more highly elevated temperatures. For these cases, more expensive and custom containments are typically required as shown in Fig. 19.

Thermomechanical fatigue (TMF) differs from the fatigue testing described earlier since the temperature of the specimen is programmed to vary in a precise manner relative to the mechanical loading. The challenge with this type of testing includes (a) achieving and (b) controlling the thermal environment required. The limiting factor is not usually with heating as induction furnaces, for instance, can heat a sample very rapidly in a uniform manner. The cycling rate is typically controlled by cooling and concerns regarding large thermal gradients exist if cooling occurs too quickly. Recent work within ASTM has generated a draft standard for TMF testing that is currently undergoing balloting.

Acknowledgments

The author would like to graciously acknowledge the wisdom and lessons gained from working closely with two highly experienced colleagues and mentors at Southwest Research Institute. Jack FitzGerald taught me that often the simplest approach, the one that seemed too elementary and often at odds with engineering education, was always the best approach when faced with something unusual or

unknown in the laboratory. Furthermore, Andy Nagy exhibited incredible creativity by taking half-baked, malformed ideas and transforming them into real devices that functioned flawlessly in the test lab. Both of these consummate professionals, now deceased, provided excellent role models and mentors for the development of an inexperienced test engineer. Finally, gratitude is extended to FTA Inc, Instron, and MTS for supplying some of the photographs used herein.

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CHAPTER 20

PLASTICS TESTING

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Not too long ago, the concept of testing was merely an afterthought of the procurement process. But now, with the advent of science and technology, the concept of testing is an integral part of research and development, product design, and manufacturing. The question that is often asked is "why test?" The answer is simple. Times have changed. The manner in which we do things today is different. The emphasis is on automation, high production, and cost reduction. There is a growing demand for intricately shaped, high-tolerance parts. Consumer awareness, a subject totally ignored by the manufacturers once upon a time, is now a major area of concern. Along with the requirements, our priorities have also changed. While designing a machine or a product, the first order of priority in most cases is safety and health. Manufacturers and suppliers are now required to meet a variety of standards and specifications. Obviously, relying merely on past experience and quality of workmanship is simply not enough. The following are some of the major reasons for testing:

1. To prove design concepts
2. To provide a basis for reliability
3. Safety

Handbook of Materials Selection. Edited by Myer Kutz

4. Protection against product liability suits
5. Quality control
6. To meet standards and specifications
7. To verify the manufacturing process
8. To evaluate competitors' products
9. To establish a history for new materials

In the last two decades, just about every manufacturer has turned to plastics to achieve cost reduction, automation, and high yield. The lack of history along with the explosive growth and diversity of polymeric materials has forced the plastics industry into placing extra emphasis on testing and developing a wide variety of testing procedures. Through the painstaking efforts of various standards organizations, material suppliers, and mainly the numerous committees of the American Society for Testing and Materials (ASTM), over 10,000 different test methods have been developed.

The need for developing standard test methods specifically designed for plastic materials originated for two main reasons. Initially, the properties of plastic materials were determined by duplicating the test methods developed for testing metals and other similar materials. The Izod impact test, for example, was derived from the manual for testing metals. Because of the drastically different nature of plastic materials, the test methods often had to be modified. As a result, a large number of nonstandard tests were written by various parties. As many as 8–10 distinct and separate test methods were written to determine the same property. Such practice created total chaos among the developers of the raw materials, suppliers, design engineers, and ultimate end users. It became increasingly difficult to keep up with various test methods as well as to comprehend the real meaning of reported test values. The standardization of test methods acceptable to everyone solved the problem of communication between developers, designers, and end users, allowing them to speak a common language when comparing the test data and results.

In spite of the standardization of various test methods, we still face the problem of comprehension and interpretation of the test data by an average person in the plastics industry. This is due to the complex nature of the test procedures and the number of tests and testing organizations. The key in overcoming this problem is to develop a thorough understanding of what the various tests mean and the significance of the result to the application being considered. Unfortunately, the plastics industry has placed more emphasis on *how* and not enough on *why*, which obviously is more important from the standpoint of comprehension of the test results and understanding the true meaning of the values. The lack of understanding of the real meaning of heat deflection temperature, which is often interpreted as the temperature at which a plastic material will sustain static or dynamic load for a long period, is one such classic example of misinterpretation. In the chapters to follow, we concentrate on the significance, interpretation, and limitations of physical property data and test procedures. Finally, a word of caution: It is extremely important to understand that the majority of physical property tests are subject to rather large errors. As a general rule, the error of testing should be considered $\pm 5\%$. Some tests are more precise than others. Such testing errors come about from three major areas: one from the basic test itself, the second from the operators conducting the tests, and the third from the variations in the test specimens. While evaluating the test data and making decisions based on test data, one must consider the error factor to make certain that a valid difference in the test data exists.

1 MECHANICAL PROPERTIES

The mechanical properties, among all the properties of plastic materials, are often the most important properties because virtually all service conditions and the majority of end-use applications involve some degree of mechanical loading. Nevertheless, these properties are the least understood by most design engineers. The material selection for a variety of applications is quite often based on mechanical properties such as tensile strength, modulus, elongation, and impact strength. These values are normally derived from the technical literature provided by material suppliers. More often than not, too much emphasis is placed on comparing the published values of different types and grades of plastics and not enough on determining the true meaning of the mechanical properties and their relation to end-use requirements. In practical applications, plastics are seldom, if ever, subjected to a single, steady deformation without the presence of other adverse factors such as environment and temperature. Since the published values of the mechanical properties of plastics are generated from tests conducted in a laboratory under standard test conditions, the danger of selecting and specifying a material from these values is obvious. A thorough understanding of mechanical properties, tests employed to determine such properties, and the effect of adverse conditions on mechanical properties over a long period is extremely important.

1.1 Tensile Tests (ASTM D638, ISO 527-1)

Tensile elongation and tensile modulus measurements are among the most important indications of strength in a material and are the most widely specified properties of plastic materials. Tensile test, in a broad sense, is a measurement of the ability of a material to withstand forces that tend to pull it apart and to determine to what extent the material stretches before breaking. Tensile modulus, an indication of the relative stiffness of a material, can be determined from a stress–strain diagram. Different types of plastic materials are often compared on the basis of tensile strength, elongation, and tensile modulus data. Many plastics are very sensitive to the rate of straining and environmental conditions. Therefore, the data obtained by this method cannot be considered valid for applications involving load-time scales or environments widely different from this method. The tensile property data are more useful in preferential selection of a particular type of plastic from a large group of plastic materials and such data are of limited use in actual design of the product. This is because the test does not take into account the time-dependent behavior of plastic materials.

The tensile testing machine of a constant-rate-of-crosshead movement is used. It has a fixed or essentially stationary member, carrying one grip and a movable member carrying a second grip. Self-aligning grips employed for holding the test specimen between the fixed member and the movable member prevent alignment problems. A controlled velocity drive mechanism is used. Some of the commercially available machines use a closed-loop servo-controlled drive mechanism to provide a high degree of speed accuracy. A load-indicating mechanism capable of indicating total tensile load with an accuracy of ± 1% of the indicated value or better is used. Lately, the inclination is toward using digital-type load indicators, which are easier to read than the analog-type indicators. An extension indicator, commonly known as the extensometer, is used to determine the distance between two designated points located within the gauge length of the test specimen as the specimen is stretched. Figure 1 shows a commercially available tensile testing machine. The advent of new microprocessor technology has virtually eliminated time-consuming manual calculations. Stress, elongation, modulus, energy, and statistical calculations are performed automatically and presented on a visual display or hard-copy printout at the end of the test.

1.2 Flexural Properties (ASTM D790, ISO 178)

The stress–strain behavior of polymers in flexure is of interest to a designer as well as a polymer manufacturer. Flexural strength is the ability of the material to withstand bending forces applied perpendicular to its longitudinal axis. The stresses induced due to the flexural load are a combination of compressive and tensile stresses. This effect is illustrated in Fig. 2. Flexural properties are reported and calculated in terms of the maximum stress and strain that occur at the outside surface of the test bar. Many polymers do not break under flexure even after a large deflection that makes determination of the ultimate flexural strength impractical for many polymers. In such cases, the common practice is to report flexural yield strength when the maximum strain in the outer fiber of the specimen has reached 5%. For polymeric materials that break easily under flexural load, the specimen is deflected until a rupture occurs in the outer fibers.

There are several advantages of flexural strength tests over tensile tests. If a material is used in the form of a beam and if the service failure occurs in bending, then a flexural test is more relevant for design or specification purposes than a tensile test, which may give a strength value very different from the calculated strength of the outer fiber in the bent beam. The flexural specimen is comparatively easy to prepare without residual strain. The specimen alignment is also more difficult in tensile tests. Also, the tight clamping of the test specimens creates stress concentration points. One other advantage of the flexural test is that at small strains, the actual deformations are sufficiently large to be measured accurately.

There are two basic methods that cover the determination of flexural properties of plastics. Method 1 is a three-point loading system utilizing center loading on a simple supported beam. A bar of rectangular cross section rests on two supports and is loaded by means of a loading nose midway between the supports. The maximum axial fiber stresses occur on a line under the loading nose. A closeup of a specimen in the testing apparatus is shown in Fig. 3. This method is especially useful in determining flexural properties for quality control and specification purposes.

Method 2 is a four-point loading system utilizing two load points equally spaced from their adjacent support points, with a distance between load points of one-third of the support span. In this method, the test bar rests on two supports and is loaded at two points (by means of two loading noses), each an equal distance from the adjacent support point. Method 2 is very useful in testing materials that do not fail at the point of maximum stress under a three-point

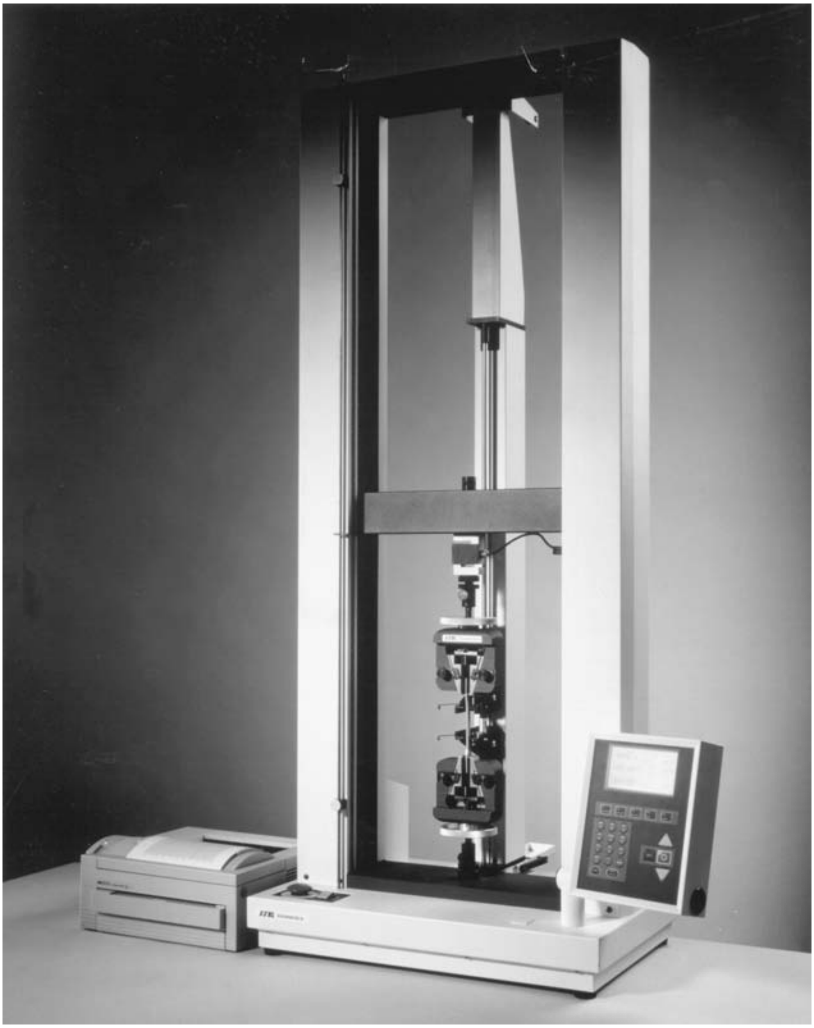


Fig. 1 Tensile testing machine. (Courtesy Tinius Olsen Corporation)

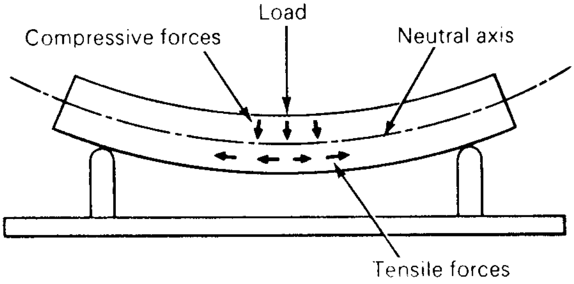


Fig. 2 Forces involved in bending a simple beam. (Reprinted by permission of McGraw-Hill Company)

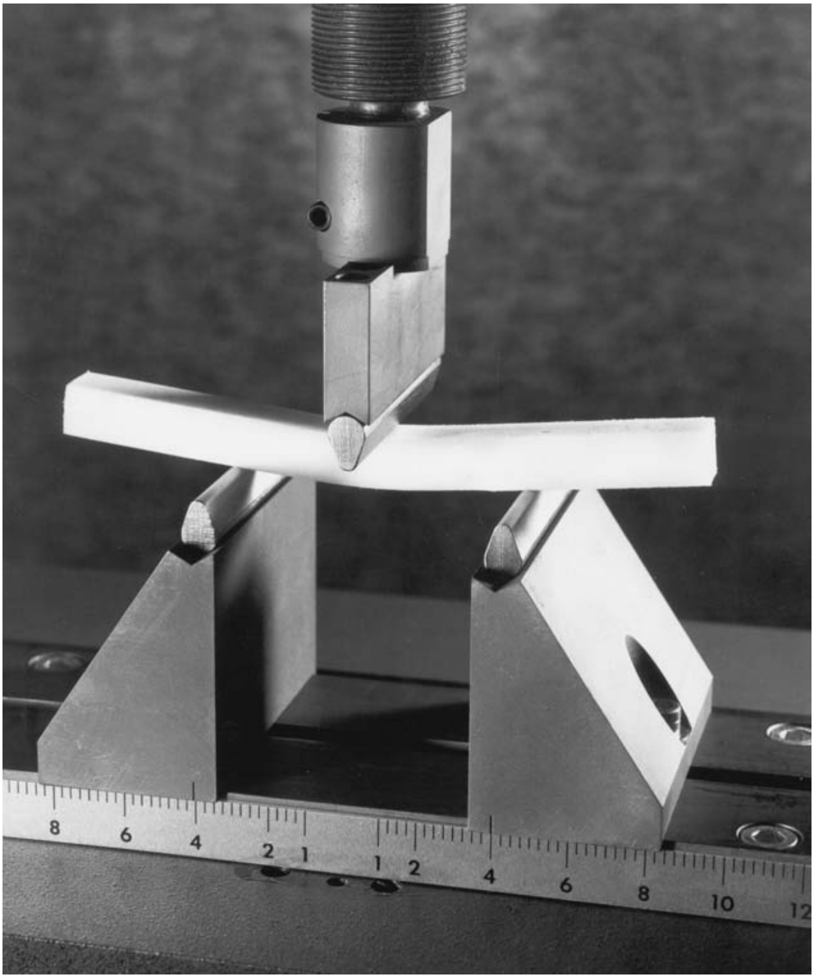


Fig. 3 Close-up of a specimen shown in flexural testing apparatus. (Courtesy DuPont Company)

loading system. The maximum axial fiber stress occurs on a line under the loading nose.

Either method can be used with the two procedures. Procedure A is designed principally for materials that break at comparatively small deflections. Procedure B is designed particularly for those materials that undergo large deflections during testing. The basic difference between the two procedures is the strain rate, procedure A being 0.01 in./in./min, and procedure B being 0.10 in./in./min.

Modulus of Elasticity (Flexural Modulus)

The flexural modulus is a measure of the stiffness during the first or initial part of the bending process. This value of the flexural modulus is, in many cases, equal to the tensile modulus.

The flexural modulus is represented by the slope of the initial straight-line portion of the stress–strain curve and is calculated by dividing the change in stress by the corresponding change in strain.

1.3 Creep Properties

Today, plastics are used in applications that demand high performance and extreme reliability. Many components, conventionally made of metals, are now made of plastics. The pressure is put on the design engineer to design the plastic products more efficiently. An increasing number of designers have now recognized the importance of thoroughly understanding the behavior of plastics under long-term load and varying temperatures. Such behavior is described in terms of creep properties.

When a plastic material is subjected to a constant load, it deforms quickly to a strain roughly predicted by its stress–strain modulus, and then continues to deform slowly with time indefinitely or until rupture or yielding causes failure. This phenomenon of deformation under load with time is called creep. All plastics creep to a certain extent. The degree of creep depends upon several factors, such as type of plastic, amount of load, temperature, and time.

The short-term stress–strain data is of little practical value in actual designing the part, since such data does not take into account the effect of long-term loading on plastics. Creep behavior varies considerably among types of plastics; however, under proper stress and temperature conditions, all plastics will exhibit a characteristic type of creep behavior. One such generalized creep curve is shown in Fig. 4. The total creep curve is divided into four continuous stages.

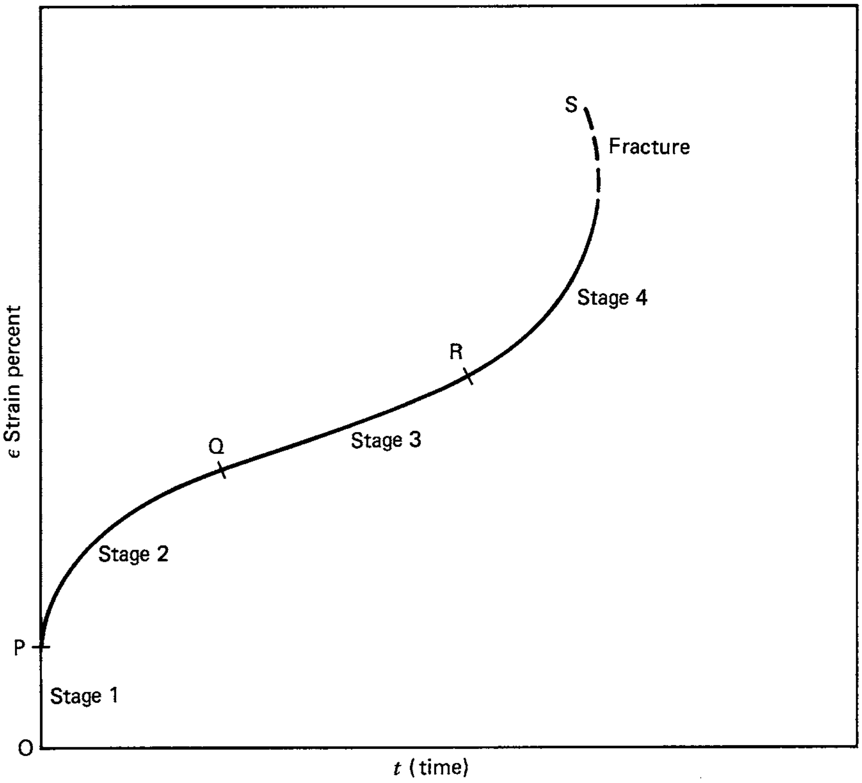


Fig. 4 Generalized creep curve

The first stage (OP) represents the instantaneous elastic deformation. This initial strain is the sum of the elastic and plastic strain. The first stage is followed by the second stage (PQ) in which strain occurs rapidly but at a decreasing rate. This stage, where creep rate decreases with time, is sometimes referred to as creep or primary creep. The straight portion of the curve (QR) is characterized by a constant rate of creep. This process is called ‘‘cold flow.’’ The final stage (RS) is marked by increase in creep rate until the creep fracture occurs.

If the applied load is released before the creep rupture occurs, an immediate elastic recovery, substantially equal to elastic deformation followed by a period of slow recovery is observed. The material in most cases does not recover to the original shape and a permanent set remains. The magnitude of the permanent set depends upon length of time, amount of stress applied, and temperature.

The creep values are obtained by applying constant load to the test specimen in tension, compression, or flexure and measuring the deformation as a function of time. The values are most commonly referred to as tensile creep, compressive creep, and flexural creep.

Tensile Creep

Tensile creep measurements are made by applying the constant load to a tensile test specimen and measuring its extension as a function of time. The extension measurement can be carried out several different ways. The simplest way is to make two gauge marks on the tensile specimen and measure the distance between the marks at specified time intervals. The percent creep strain is determined by dividing the extension by initial gauge length and multiplying by 100. The percent creep strain is plotted against time to obtain a tensile creep curve. The tensile stress values are also determined at specified time intervals to facilitate plotting a stress rupture curve. The more accurate measurements require the use of a strain gauge, which is capable of measuring and amplifying small changes in length with time and directly plotting them on a chart paper. Figure 5 illustrates a typical setup for tensile creep testing. The test is also carried out at different stress levels and temperatures to study their effect on tensile creep properties.

Flexural Creep

Flexural creep measurements are also made by applying a constant load to the standard flexural test specimen and measuring its deflection as a function of time. A typical test setup for measuring creep in flexure is shown in Fig. 6. As illustrated, the deflection of the specimen at midspan is measured using a dial indicator gauge. The electrical resistance gauges may also be used in place of a dial indicator. The deflections of the specimen are measured at a predetermined time interval.

1.4 Stress Relaxation

Stress relaxation is defined as a gradual decrease in stress with time, under a constant deformation (strain). This characteristic behavior of the polymers is studied by applying a fixed amount of deformation to a specimen and measuring the load required to maintain it as a function of time. This is in contrast to creep measurement, where a fixed amount of load is applied to a specimen and resulting deformation is measured as a function of time.

Fig. 5 Typical test setup for tensile creep testing. (Courtesy Applied Test Systems Inc.)

Fig. 6 Flexural creep testing. (Courtesy Ceast U.S.A. Inc.)

Stress relaxation behavior of the polymers has been overlooked by many design engineers and researchers, partly because the creep data is much easier to obtain and is readily available. However, many practical applications dictate the use of stress relaxation data. For example, extremely low-stress relaxation is desired in the case of a threaded bottle closure, which may be under constant strain for a long period. If the plastic material used in the closures shows an excessive decrease in stress under this constant deformation, the closures will eventually fail. Similar problems can be encountered with metal inserts in molded plastics, belleville or multiple cantilever springs used in cameras, appliances, and business machines.

Stress relaxation measurements can be carried out using a tensile testing machine such as that described earlier in this chapter. However, the use of such a machine is not always practical because the stress relaxation test ties up the machine for a long period of time. The equipment for a stress relaxation test must be capable of measuring very small elongation accurately, even when applied at high speeds. Many sophisticated pieces of equipment now employ a strain gauge or a differential transformer along with a chart recorder capable of plotting stress as a function of time. A typical stress–time curve is schematically plotted in Fig. 7. At the beginning of the experiment, the strain is applied to the specimen at a constant rate to achieve the desired elongation. Once the specimen reaches the desired elongation, the strain is held constant for a predetermined amount of time. The stress decay, which occurs due to stress relaxation, is observed as a function of time. If a chart recorder is not available, the stress values at different time intervals are recorded and the results are plotted to obtain a stress versus time curve. The stress relaxation experiment is often carried out at various levels of temperature and strain. The stress data obtained from the stress relaxation experiment can be converted to a more meaningful apparent modulus data by simply dividing stress at a particular time by the applied strain. The curve may be replotted to represent apparent modulus as a function of time. The use of logarithmic coordinates further simplifies the stress relaxation data by

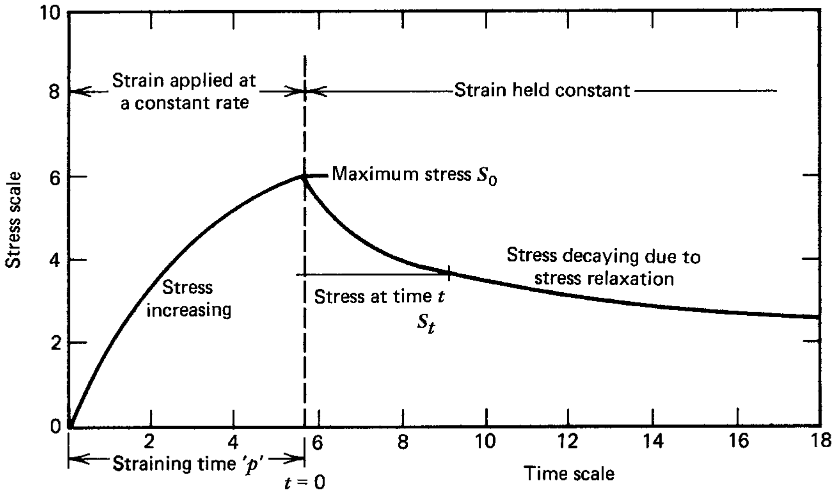


Fig. 7 Stress–time curve. (Courtesy Instron Corporation)

allowing us to use standard extrapolation methods such as the one used in creep experiments.

1.5 Impact Properties

The impact properties of the polymeric materials are directly related to the overall toughness of the material. Toughness is defined as the ability of the polymer to absorb applied energy. The area under the stress–strain curve is directly proportional to the toughness of a material. Impact strength is a measure of toughness. The higher the impact strength of a material, the higher the toughness and vice versa. Impact resistance is the ability of a material to resist breaking under a shock loading or the ability to resist the fracture under stress applied at high speed.

The theory behind toughness and brittleness of the polymers is very complex and therefore difficult to understand. The molecular flexibility plays an important role in determining the relative brittleness or toughness of the material. For example, in stiff polymers such as polystyrene and acrylics, the molecular segments are unable to disentangle and respond to the rapid application of mechanical stress and the impact produces brittle failure. In contrast, flexible polymers such as plasticized vinyls have high-impact strength due to the ability of the large segments of molecules to disentangle and respond rapidly to mechanical stress.

Impact properties of the polymers are often modified simply by adding an impact modifier such as butadiene rubber or certain acrylic compounds. The addition of a plasticizer also improves the impact strength at the cost of rigidity. Material such as nylon, which has relatively fair impact strength, can be oriented by aligning the polymer chains to improve the impact strength substantially. Another way to improve the impact strength is to use fibrous fillers that appear to act as stress transfer agents.

Most polymers, when subjected to the impact loading, seem to fracture in a characteristic fashion. The crack is initiated on a polymer surface due to the impact loading. The energy to initiate such a crack is called the crack initiation energy. If the load exceeds the crack initiation energy, the crack continues to propagate. A complete failure occurs when the load has exceeded the crack propagation energy. Thus, both crack initiation and crack propagation contribute to the measured impact strength. There are basically four types of failures encountered due to the impact load.

Brittle Fracture. In this type of failure the part fractures extensively without yielding. A catastrophic mechanical failure such as the one in the case of general-purpose polystyrene is observed.

Slight Cracking. The part shows evidence of slight cracking and yielding without losing its shape or integrity.

Yielding. The part actually yields showing obvious deformation and stress whitening but no cracking takes place.

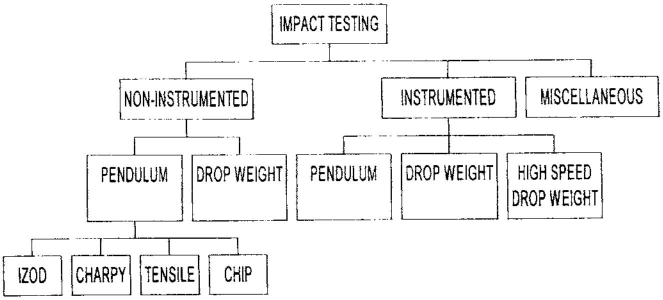
Ductile Failure. This type of failure is characterized by a definite yielding of material along with cracking. Polycarbonate is considered a ductile material.

The distinction between the four types of failures is not very clear and some overlapping is quite possible.

Impact strength is one of the most widely specified mechanical properties of the polymeric materials. However, it is also one of the least understood properties. Predicting the impact resistance of plastics still remains one of the most troublesome areas of product design. One of the problems with some earlier Izod and Charpy impact tests was that the tests were adopted by the plastic industry from metallurgists. The principles of impact mechanisms as applied to metals do not seem to work satisfactorily with plastics because of the plastics' complex structure.

Types of Impact Tests

In the 1980s and 1990s, a tremendous amount of time and money have been spent on research and development of various types of impact tests by organizations throughout the world. Attempts have been made to develop different sizes and shapes of specimens as well as impact testers. The specimens have been subjected to a variety of impact loads including tensile, compression, bending, and torsion impacts. Impact load has been applied using everything from a hammer, punches, and pendulums to falling balls and bullets. Unfortunately, very little correlation exists, if any, between the types of tests developed so far. Numerous technical papers and articles have been written on the subject of the advantages of one method over the other. To this date, no industrywide consensus exists regarding an ideal impact test method. In this chapter, an attempt is made to discuss as many types of impact tests as possible along with the respective advantages and limitations of each test. Impact testing is divided into three major classes and subdivided into several classes as follows:



Pendulum Impact Test: IZOD–CHARPY (ASTM D-256, ISO 179). The objective of the Izod–Charpy impact test is to measure the relative susceptibility of a standard test specimen to the pendulum-type impact load. The results are expressed in terms of kinetic energy consumed by the pendulum in order to break the specimen. The energy required to break a standard specimen is actually the sum of energies needed to deform it, to initiate its fracture, and to propagate the fracture across it, and the energy expended in tossing the broken ends of the specimen. This is called the *toss factor.* The energy lost through the friction and vibration of the apparatus is minimal for all practical purposes and usually neglected.

The specimen used in Izod test must be notched. The reason for notching the specimen is to provide a stress concentration area that promotes a brittle rather than a ductile failure. A plastic deformation is prevented by such type of notch in the specimen. The impact values are seriously affected because of the notch sensitivity of certain types of plastic materials.

The Izod test requires a specimen to be clamped vertically as a cantilever beam. The specimen is struck by a swing of a pendulum released from a fixed distance from the specimen clamp. A similar setup is used for the Charpy test except for the positioning of the specimen. In the Charpy method, the specimen is supported horizontally as a simple beam and fractured by a blow delivered in the middle by the pendulum. The obvious advantage of the Charpy test over the Izod test is that the specimen does not have to be clamped and, therefore, it is free of variations in clamping pressures.

Apparatus and Test Specimens. The testing machine consists of a heavy base with a vise for clamping the specimen in place during the test. In most cases, the vise is designed so that the specimen can be clamped vertically for the Izod test or positioned horizontally for the Charpy test without making any changes. A pendulum-type hammer with an antifriction bearing is used. Additional weights may be attached to the hammer for breaking tougher specimens. The pendulum is connected to a pointer and a dial mechanism that indicates the excess energy remaining in a pendulum after breaking the specimen. The dial is calibrated to read the impact values directly in in.-lb or ft-lb. A hardened steel striking nose is attached to the pendulum. The Izod and Charpy tests use different types of striking noses. A detailed list of requirements is discussed in the ASTM standards book. Figure 8 illustrates a typical pendulum-type impact testing machine. The test specimens can be prepared either by molding or cutting them from a sheet. Izod test specimens are $2\frac{1}{2} \times \frac{1}{2} \times \frac{1}{8}$ in. size. The most common specimen thickness is $\frac{1}{8}$-in. but $\frac{1}{4}$ in. is preferred since it is less susceptible to bending and crushing. A notch is cut into a specimen very carefully by a milling machine or a lathe. The recommended notch depth is 0.100-in.

Test Procedures

Izod Test. The test specimen is clamped into position so that the notched end of the specimen is facing the striking edge of the pendulum. The pendulum hammer is released, allowed to strike the specimen, and swing through. If the specimen does not break, more weights are attached to the hammer and the test is repeated until failure is observed. The impact values are read directly in in.-lbf or ft-lbf from the scale. The impact strength is calculated by dividing the impact values obtained from the scale by the thickness of the specimen. For example, if a reading of 2 ft-lbf is obtained using an $\frac{1}{8}$-in.-thick specimen, the impact value would be 16 ft-lbf/in. of notch. The impact values are always calculated on the basis of 1-in.-thick specimens even though much thinner specimens are usually used. The reversed notched impact strength is obtained by reversing the position of a notched specimen in the vise. In this case, the notch is subjected to compressive rather than tensile stresses during impact. As discussed earlier in this chapter, the energy required to break a specimen is the sum of the energies needed to deform it, initiate and propagate the fracture, and toss the broken end (toss factor).

Falling-Weight Impact Test. The falling-weight impact test, also known as the drop impact test or the variable-height impact test, employs a falling weight. This falling weight may be a tup with a conical nose, a ball, or a ball-end dart.

Fig. 8 Pendulum impact tester. (Courtesy Tinius Olsen Company)

The energy required to fail the specimen is measured by dropping a known weight from a known height onto a test specimen. The impact energy is normally expressed in ft-lb and is calculated by multiplying the weight of the projectile by the drop height.

The biggest advantage of the falling-weight impact test over the pendulum impact test or high-rate tension test is its ability to duplicate the multidirectional impact stresses that a part would be subjected to in actual service. The other obvious advantage is the flexibility to use specimens of different sizes and shapes, including an actual part. Unlike the notched Izod impact test, which measures the notch sensitivity of the material and not the material toughness, falling-weight impact tests introduce polyaxial stresses into the specimen and measure the toughness. The variations in the test results due to the fillers and reinforcements, clamping pressure, and material orientation are virtually eliminated in the falling-weight impact test. This type of test is also very suitable for determining the impact resistance of plastic films, sheets, and laminated materials.

Three basic ASTM tests are commonly used depending upon the application:

ASTM D5420. Impact resistance of flat rigid plastic specimen by means of a falling weight

ASTM D1709. Impact resistance of plastic film by the free-falling dart method

ASTM D2444. Test for impact resistance of thermo-plastic pipe and fittings by means of a tup

Drop Impact Test. This falling-weight impact test is primarily designed to determine the relative ranking of materials according to the energy required to break flat rigid plastic specimens under various conditions of impact of a striker impacted by a falling weight. A free-falling weight, or a tup, is used to determine the impact resistance of the material.

Many different versions of test equipment exist today. They all basically operate on the same principle. Figure 9 illustrates one such typical commercially available testing machine. It consists of a cast aluminum base, a slotted vertical guide tube, a round-nose striker and striker holder, an 8-lb weight, a die and die

Fig. 9 Drop impact tester. (Courtesy Byk-Gardner USA)

support, and a sample platform. The sample platform is used to position a sheet of desired thickness for impact testing. The die is removable from the base in order that the actual parts of complex shapes can be placed onto the base and impact tested.

The test is carried out by raising the weight to a desired height manually or automatically with the use of a motor-driven mechanism and allowing it to fall freely onto the other side of the striker. The striker transfers the impact energy to the flat test specimen positioned on a cylindrical die or a part lying on the base of the machine. The kinetic energy possessed by the falling weight at the instant of impact is equal to the energy used to raise the weight to the height of the drop and is the potential energy possessed by the weight as it is released. Since the potential energy is expressed as the product of weight and height, the guide tube can be marked with a linear scale representing the impact range of the instrument in in.-lb. Thus, the toughness or the impact resistance of a specimen or a part can be read directly off the calibrated scale in in.-lb. The energy loss due to the friction in the tube or due to the momentary acceleration of the punch is negligible.

An alternate method for achieving the same result utilizes an instrument that employs a free-falling dart dropped from a specified height onto a test specimen. The dart with a hemispherical head is constructed of smooth, polished aluminum or stainless steel. An electromagnetic, air-operated or other mechanical release mechanism with a centering device is used for releasing the dart. The dart is also fitted with a shaft long enough to accommodate removable incremental weights. A two-piece annular specimen clamp is used to hold the specimen.

Instrumented Impact Testing. One of the biggest drawbacks of the conventional impact test methods is that it provides only one value—the total impact energy—and nothing else. The conventional tests cannot provide additional information on the ductility, dynamic toughness, fracture, and yield loads or the behavior of the specimen during the entire impact event.

This effectively limits the application of noninstrumented impact test methods to quality control and material ranking. Instrumented impact testers are generally suited for research and development as well as advance quality control.

Instrumented impact testers measure force continuously while the specimen is penetrated. The resulting data can be used to determine type of failure and maximum load, in addition to the amount of energy required to fracture the specimen. One of the most common type of failures occurring from ductile to brittle transition at low temperatures can only be observed by studying the load–energy–time curve. The fracture mode of a plastic is sensitive to the changes in temperature, and can change abruptly at or near the materials transition temperature. Manufacturers of plastic automotive components routinely test materials at low temperatures (−20 to −40°F) to assure that they will not become brittle in cold weather. By studying the shape of the load–time or load–deflection curve, the type of failure can be analyzed, and important information about its performance in service can be gathered. The new piezoelectric-equipped strikers offer increased sensitivity, opening the doors for testing a whole new range of materials. Applications involving light-weight products such as foam containers for eggs and ultra-thin films used in packaging industry can now be meaningfully tested.

All standard impact testers can be instrumented to provide a complete load and energy history of the specimen. Such a system monitors and precisely records the entire impact event, starting from the acceleration (from rest) to the initial impact and plastic bending to fracture initiation and propagation to the complete failure. The instrumentation is done by mounting the strain gauges or load cell onto the striking bit in the case of pendulum impact tester or onto the tup in the case of a drop impact tester. During the test, a fiber-optic device triggers an oscilloscope just before striking the specimen. The output of the strain gauge is recorded by the oscilloscope depicting the variation of the load applied to the specimen throughout the entire fracturing process. A complete load–time history of the entire specimen is obtained. The apparent total energy absorbed by the specimen can be calculated and plotted against time. The specimen displacement can be calculated by the double integration of the load–time curve and the load–displacement curve can be plotted. With the advent of microprocessor technology, some manufacturers are now capable of offering a unit that automatically calculates the sample displacement and provides a load–displacement curve eliminating the need for calculations. Many other useful data such as the impact rate, force and displacement at yield, and break, yield, and failure energies as well as modulus are calculated and printed out. A commercially available instrumented impact tester is shown in Fig. 10.

Interpreting Impact Data

Modern instrumented impact test machines generate a complete record of (1) force applied to the specimen versus time, (2) displacement of the impactor

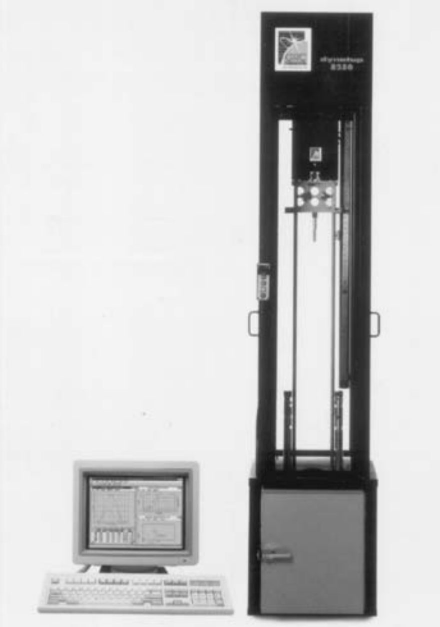


Fig. 10 Instrumented impact tester. (Courtesy Instron Corporation)

(from the point of specimen contact) versus time, (3) velocity of the impactor versus time, and (4) energy absorbed by the specimen versus time.

When plotted (Fig. 11), many details of the impact event become clearly visible in the data. Most systems also analyze the data and provide tabulated reports of critical values. Results of instrumented impact tests are interpreted differently depending on the type of material being tested and the failure criteria applied.

For homogeneous materials, four values are critical:

Maximum (yield) load is simply the highest point on the load–time curve. Often the point of maximum load corresponds to the onset of material damage or complete failure.

Energy to maximum (yield) load is the energy absorbed by the specimen up to the point of maximum load. When maximum load corresponds to failure, the energy to max load is the amount of energy the specimen can absorb before failing.

Total energy is the amount of energy the specimen absorbed during the complete test. This number may be the same as energy to maximum load when the specimen abruptly fails at the maximum load point.

Deflection to maximum (yield) load is the distance the impactor traveled from the point of impact to the point of maximum load. Figure 12 shows a typical test data.

High-Speed Impact Tests (ASTM D3763, ISO 6603-2). An ever-increasing demand for engineering plastics and the need for sophisticated and meaningful impact test methods for characterizing these materials have forced the industry into developing new high-speed impact tests. These tests not only provide the

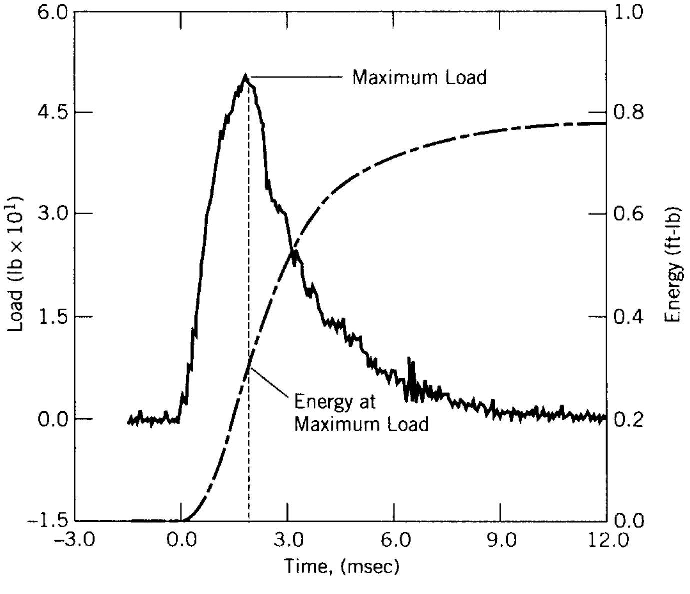
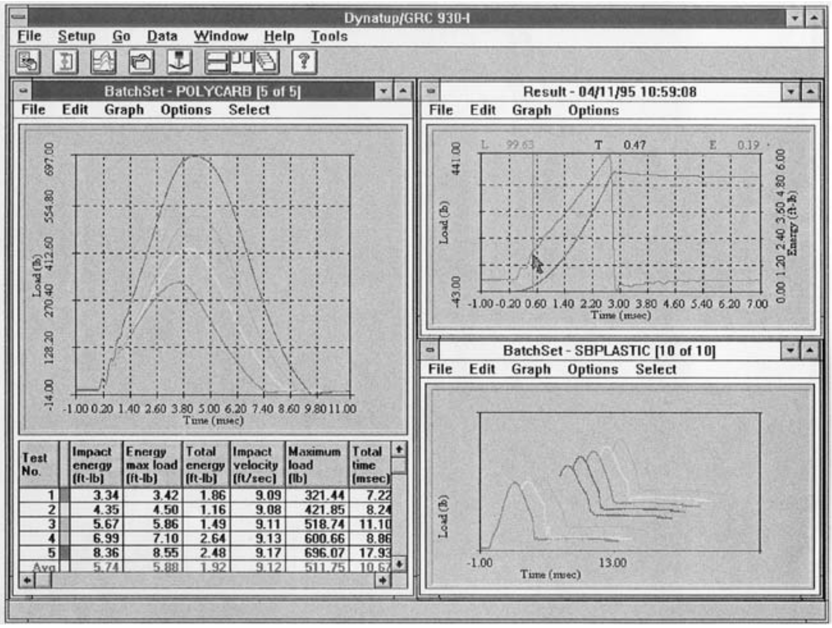


Fig. 11 Load–energy–time curve showing effect of impact on a specimen.



Test No.		Impact energy (ft-lb)	Energy max load (ft-lb)	Total energy (ft-lb)	Impact velocity (ft/sec)	Maximum load (lb)	Total time (msec)
1		3.34	3.42	1.86	9.09	321.44	7.22
2		4.35	4.50	1.16	9.08	421.85	8.24
3		5.67	5.86	1.49	9.11	518.74	11.1
4		6.99	7.10	2.64	9.13	600.66	8.8
5		8.36	8.55	2.48	9.17	696.07	17.9
Avg		5.74	5.88	1.92	9.12	511.75	10.6

Fig. 12 Typical test data obtained with instrumented impact tester. (Courtesy Instron Corporation)

basic information regarding the toughness of the polymeric materials but also provide other important data of interest, such as the load–deflection curve and the total energy absorption. The high-speed impact test overcomes the basic limitations of conventional impact testing methods as discussed previously. The rate of impact can be varied from 30 to 570,000 in./min.

High-speed impact testing has gained considerable popularity in recent years because of its ability to simulate actual impact failures at high speeds. For example, conventional impact testing methods are useless in testing to meet advanced automotive crash standards that require the impact simulation at 28 mph (30,000 in./min). High-speed impact testers are able to meet the challenge. As discussed earlier in this chapter, almost all polymers are strain-rate sensitive. Two polymers, when impact tested at one strain rate, may show similar impact strength values. The same two polymers tested at a high strain rate show a completely different set of values.

Most versatile high-speed impact testing machine is capable of testing everything from the thin film that may require as low an impact rate as 30 in./min to the plastic automotive bumper that may require a high impact rate up to 30,000 in./min. The specimen or product can be tested under a controlled environment of temperature and humidity. The equipment basically consists of a tup attached to a motor wound spring or pneumatically powered actuator along with a plunger displacement measuring system. The force is detected with a fast responding quartz load cell mounted directly on the actuator. The velocity can be set digitally from 30 to 30,000 in./min. Some type of clamp assembly to hold the specimen in place is used. The equipment can also be fitted with an environmental chamber for specialized testing. The tester is equipped with a

CRT and *x-y* plotter that automatically displays load versus displacement data. A built-in microprocessor provides more useful information, such as modulus, yield, and failure energies.

High-speed impact testers have been proven very useful in material evaluation. At low strain rates, some polymers fail in ductile manner. The same polymers appear to show brittle failure at high strain rates. The point at which this ductile-to-brittle transition takes place is of particular importance. A high-rate impact test can provide such information in a graphical form. Tests can also be carried out at different temperatures to find ductile–brittle transition points at various temperatures. Other useful applications of the high-speed impact tester include the process quality control, design evaluation, and assembly evaluation.

Abrasion Resistance Tests

The material's ability to resist abrasion is most often measured by its loss in weight when abraded with an abraser. The most widely accepted abraser in the industry is called the Taber abraser. A variety of wheels with varying degree of abrasiveness is available. The grade of "calibrase" wheel designated CS-17 with 1000-g load seems to produce satisfactory results with almost all plastics. For softer materials less abrasive wheels with smaller load on the wheels may be used. The test specimen is usually a 4-in.-diameter disk or a 4-in.^{2} plate having both surfaces substantially plane and parallel. A $\frac{1}{2}$-in.-diameter hole is drilled in the center. Specimens are conditioned employing standard conditioning practices prior to testing. To commence testing, the test specimen is placed on a revolving turntable. Suitable abrading wheels are placed on the specimen under certain set dead-weight loads. The turntable is started and an automatic counter records the number of revolutions. Most tests are carried out to at least 5000 revolutions. The specimens are weighed to the nearest milligram. The test results are reported as weight loss in mg/1000 cycles. The grade of abrasive wheel along with amount of load at which the test was carried out is always reported along with results.

Test methods such as ASTM D1044 (resistance of transparent plastic materials to abrasion) are also developed for estimating the resistance of transparent plastic materials to one kind of abrasion by measurement of its optical affects. The test is carried out in similar manner to that described above, except that 100 cycles with a 500-g load is normally used. A photoelectric photometer is used to measure the light scattered by abraded track. The percentage of the transmitted light that is diffused by the abraded specimens is reported as a test result.

Another method to study the resistance of plastic material to abrasion is by measuring the volume loss when a flat specimen is subjected to abrasion with loose abrasive or bonded abrasive on cloth or paper. This method is designated as ASTM D1242. Figure 13 illustrates a commercially available abrasion tester.

1.6 Fatigue Resistance

The behavior of materials subjected to repeated cyclic loading in terms of flexing, stretching, compressing, or twisting is generally described as fatigue. Such repeated cyclic loading eventually constitutes a mechanical deterioration and progressive fracture that leads to complete failure. Fatigue life is defined as the

Fig. 13 Abrasion tester. (Courtesy Taber Industries)

number of cycles of deformation required to bring about the failure of the test specimen under a given set of oscillating conditions.

The failures that occur from repeated application of stress or strain are well below the apparent ultimate strength of the material. Fatigue data are generally reported as the number of cycles to fail at a given maximum stress level. The fatigue endurance curve, which represents stress versus number of cycles to failure, also known as *S–N* curve, is generated by testing a multitude of specimens under cyclic stress, each one at different stress levels. At high-stress levels, materials tend to fail at relatively low numbers of cycles. At low stresses, the materials can be stressed cyclically for an indefinite number of times and the failure point is virtually impossible to establish. This limiting stress below which material will never fail is called the fatigue endurance limit. The fatigue endurance limit can also be defined as the stress at which the *S–N* curve becomes asymptotic to the horizontal (constant stress) line. For most polymers, the fatigue endurance limit is between 25 and 30% of the static tensile strength. The fatigue resistance data are of practical importance in the design of gears, tubing, hinges, parts on vibrating machinery, and pressure vessels under cyclic pressures.

Two basic types of tests have been developed to study the fatigue behavior of plastic materials:

1. Flexural fatigue test
2. Tensile fatigue test

Flexural Fatigue Test (ASTM D671)

The ability of a material to resist deterioration from cyclic stress is measured in this test by using a fixed cantilever-type testing machine capable of producing a constant amplitude of force on the test specimen each cycle. The main feature of a fatigue testing machine is an unbalanced, variable eccentric, mounted on a shaft that is rotated at a constant speed by a motor. This unbalanced movement of an eccentric produces alternating force. The specimen is held as a cantilever beam in a vice at one end and bent by a concentrated load applied through a yoke fastened to the opposite end. A counter is used to record the number of cycles along with a cutoff switch to stop the machine when the specimen fails. A typical commercially available fatigue testing machine is illustrated in Fig. 14. The test specimen of two different geometries are used. If machined specimens are used, care must be taken to eliminate all scratches and tool marks from the specimens. Molded specimen must be stress relieved before using.

The test is carried out by first determining the complementary mass and effective mass of the test specimen. The load required to produce the desired stress is calculated from these values. The number of cycles required to produce failure is determined. The test is repeated at varying stress levels. A curve of stress versus cycles to failure (*S–N* diagram) is plotted from the test results.

Tensile Fatigue Test

Unlike the flexural fatigue test, which uses the constant deflection (strain) principle, the tensile fatigue test is conducted under constant-load (stress) conditions. The specimen is dumbbell-shaped, about 2 in. long with a cylindrical cross section.

The test is conducted by mounting both ends of the dumbbell specimen in the testing machine. The specimen is rotated between two spindles, and stress in the form of tension and compression is applied. The specimen is subjected to the number of cycles of stress specified or until fracture occurs.

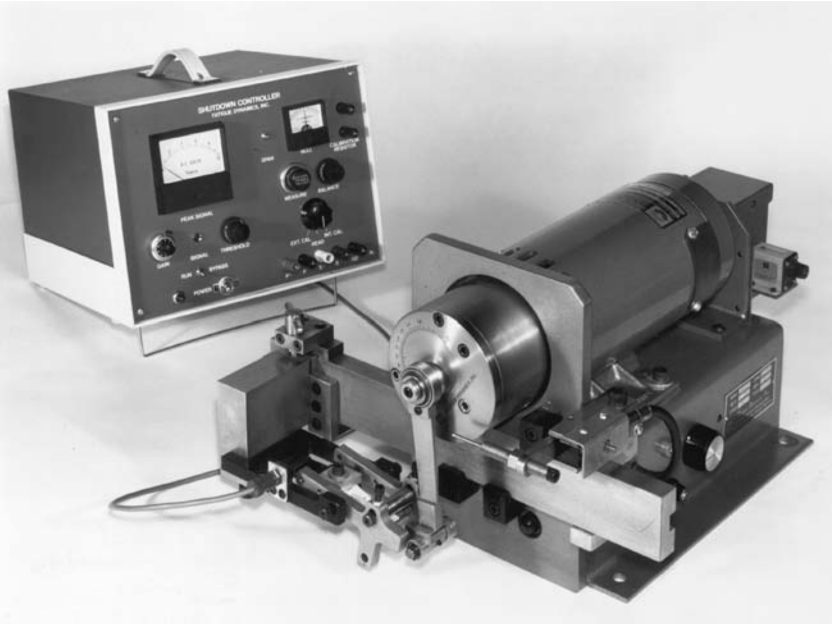


Fig. 14 Flexural fatigue tester. (Courtesy Fatigue Dynamics, Inc.)

1.7 Hardness Tests

Hardness is defined as the resistance of a material to deformation, particularly permanent deformation, indentation, or scratching. Hardness is purely a relative term and should not be confused with wear and abrasion resistance of plastic materials. Polystyrene, for example, has a high Rockwell hardness value but a poor abrasion resistance. Hardness test can differentiate relative hardness of different grades of a particular plastic. However, it is not valid to compare hardness of various types of plastics entirely on the basis of one type of test, since elastic recovery along with hardness is involved. The test is further complicated by a phenomenon such as creep. Many tests have been devised to measure hardness. Since plastic materials vary considerably with respect to hardness, one type of hardness test is not applicable to cover the entire range of hardness properties encountered. Two of the most commonly used hardness tests for plastics are the Rockwell hardness test and the Durometer hardness test. Rockwell hardness is used for relatively hard plastics such as acetals, nylons, acrylics, and polystyrene. For softer materials such as flexible polyvinyl chloride (PVC), thermoplastic rubbers, and polyethylene, Durometer hardness is often used. The typical hardness values of some common plastic materials are listed for comparison in Table 1.

Rockwell Hardness (ASTM D785)

The Rockwell hardness test measures the net increase in depth impression as the load on an indenter is increased from a fixed minor load to a major load and then returned to a minor load. The hardness numbers derived are just numbers without units. Rockwell hardness numbers are always quoted with a scale symbol representing the indenter size, load, and dial scale used. The hardness scales in order of increasing hardness are *R*, *L*, *M*, *E*, and *K* scales. The higher the number in each scale, the harder the material. There is a slight overlap of hardness scales and, therefore, it is quite possible to obtain two different dial readings on different scales for the same material. For a specific type of material,

Table 1 Typical Hardness Values of Some Common Plastic Materials

	Hardness		
	Rockwell		Durometer
Plastic material	M	R	Shore D
Acetal	94	120	
Acrylic	85–105		
Acrylonitrile butadiene styrene		75–115	
Cellulosics		30–125	
Polyphenylene oxide	80	120	
Nylon		108–120	
Polycarbonate	72	118	
High-density polyethylene			60–70
Low-density polyethylene			40–50
Polypropylene			75–85
Polystyrene (general purpose)	68–70		
Polyvinyl chloride (rigid)		115	
Polysulfone	70	120	

correlation in the overlapping regions is possible. However, due to differences in elasticity, creep, and shear characteristics between different plastics, a general correlation is not possible.

Durometer Hardness (ASTM D2240, ISO 868)

The Durometer hardness test is mostly used for measuring the relative hardness of soft materials. The test method is based on the penetration of a specified indentor forced into the material under specified conditions.

The Durometer hardness tester consists of a pressure foot, an indentor, and an indicating device. The indentor is spring loaded and the point of the indentor protrudes through the hole in the base. The test specimens are at least $\frac{1}{4}$-in. thick and can be either molded or cut from a sheet. Several thin specimens may be piled to form a $\frac{1}{4}$-in.-thick specimen but one piece specimens are preferred. The poor contact between the thin specimens may cause results to vary considerably.

The test is carried out by first placing a specimen on a hard, flat surface. The pressure foot of the instrument is pressed onto the specimen, making sure that it is parallel to the surface of the specimen. The durometer hardness is read within 1 sec after the pressure foot is in firm contact with the specimen.

Two types of durometers are most commonly used—type A and type D. The basic difference between the two types is the shape and dimension of the indentor. The hardness numbers derived from either scale are just numbers without any units. Type A durometer is used with relatively soft material while type D durometer is used with slightly harder material.

2 THERMAL PROPERTIES

Thermal properties of plastic materials are equally as important as mechanical properties. Unlike metals, plastics are extremely sensitive to changes in temperature. The mechanical, electrical, or chemical properties of plastics cannot be looked at without looking at the temperature at which the values were derived. Crystallinity has a number of important effects upon the thermal properties of a polymer. Its most general effects are the introduction of a sharp melting point and the stiffening of thermal mechanical properties. Amorphous plastics, in contrast, have a gradual softening range. Molecular orientation also has a significant effect on thermal properties. Orientation tends to decrease dimensional stability at higher temperatures. The molecular weight of the polymer affects the low-temperature flexibility and low-temperature brittleness. Many other factors such as intermolecular bonding, cross-linking, and copolymerization all have a considerable effect on thermal properties. From the above discussion, it is very clear that the thermal behavior of polymeric materials is rather complex. Therefore, in designing a plastic part or selecting a plastic material from the available thermal property data, one must thoroughly understand the short-term as well as the long-term effect of temperature on properties of that plastic material.

2.1 Tests for Elevated Temperature Performance

Designers and material selectors of plastic products constantly face the challenge of selecting a suitable plastic for elevated temperature performance. The difficulty arises due to the varying natures and capabilities of various types and grades of plastics at elevated temperatures. Many factors are considered when selecting a plastic for a high-temperature application. The material must be able to support a design load under operating conditions without objectionable creep or distortion. The material must not degrade or lose necessary additives that will cause drastic reduction in the physical properties during the expected service life.

All the properties of plastic materials are not affected in a similar manner by elevating temperature. For example, electrical properties of a particular plastic may show only a moderate change at elevated temperatures, while the mechanical properties may be reduced significantly. Also, since the properties of plastic materials vary with temperature in an irregular fashion, they must be looked at as a function of temperature in order to obtain more meaningful information. From the foregoing, it is quite clear that a single maximum-use temperature that will apply to all the important properties in high-temperature applications is simply not possible.

One of the most important considerations while studying the performance of plastics at elevated temperatures is the dependence of key properties such as modulus, strength, chemical resistance, and environmental resistance on time. Therefore, the short-term heat resistance data alone is not adequate for designing and selecting materials that require long-term heat resistance. For the sake of convenience and simplicity, we divide the elevated temperature effects into two categories:

1. Short-term effects
   - **a.** Heat deflection temperature
   - **b.** Vicat softening temperature
   - **c.** Torsion pendulum
2. Long-term effects
   - **a.** Long-term heat resistance test
   - **b.** Underwriters Laboratory (UL) temperature index
   - **c.** Creep modulus/creep rupture tests

Short-Term Effects

Heat Deflection Temperature (HDT) (ASTM D648, ISO 75-1, ISO 75-2). Heat deflection temperature is defined as the temperature at which a standard test bar ($5 \times \frac{1}{2} \times \frac{1}{4}$ in.) deflects 0.010 in. under a stated load of either 66 or 264 psi. The heat deflection temperature test, also referred to as the heat distortion temperature test, is commonly used for quality control and for screening and ranking materials for short-term heat resistance. The data obtained by this method cannot be used to predict the behavior of plastic materials at elevated temperature nor can it be used in designing a part or selecting and specifying material. Heat deflection temperature is a single-point measurement and does not indicate long-term heat resistance of plastic materials. Heat distortion temperature, however, does distinguish between those materials that lose their rigidity over a narrow temperature range and those that are able to sustain light loads at high temperatures.

The apparatus for measuring heat deflection temperature consists of an enclosed oil bath fitted with a heating chamber and automatic heating controls that raise the temperature of the heat transfer fluid at a uniform rate. A cooling system is also incorporated to fast cool the heat transfer medium for conducting repeated tests. The specimens are supported on steel supports, 4 in. apart with the load applied on top of the specimen vertically and midway between the supports. The contact edges of the support and of the piece by which pressure is applied is rounded to a radius of $\frac{1}{4}$ in. A suitable deflection measurement device, such as a dial indicator, is normally used. A mercury thermometer is used for measuring temperature. The unit is capable of applying 66 or 264 psi fiber stress on specimens by means of a dead weight. A commercially available heat deflection measuring device with a closeup of a specimen holder is illustrated in Fig. 15.

More recently, automatic heat deflection temperature testers have been developed. These testers typically replace conventional temperature and deflection measuring devices with more sophisticated electronic measuring devices with digital read-out system and a chart recorder that prints out the results. Such an automatic apparatus eliminates the need for the continuous presence of an operator and thereby minimizes operator-related errors.

The test specimens consist of test bars 5 in. in length, $\frac{1}{2}$ in. in depth by any width from $\frac{1}{8}$ to $\frac{1}{2}$ in. The test bars may be molded or cut from extruded sheet as long as they have smooth, flat surfaces and are free from excessive sink marks or flash. The specimens are conditioned employing standard conditioning procedures.

The specimen is positioned in the apparatus along with the temperature and deflection measuring devices and the entire assembly is submerged into the oil bath kept at room temperature. The load is applied to a desired value (66 or 264 psi fiber stress). Five minutes after applying the load, the pointer is adjusted to zero and the oil is heated at the rate of 2 ± 0.2°C/min. The temperature of the

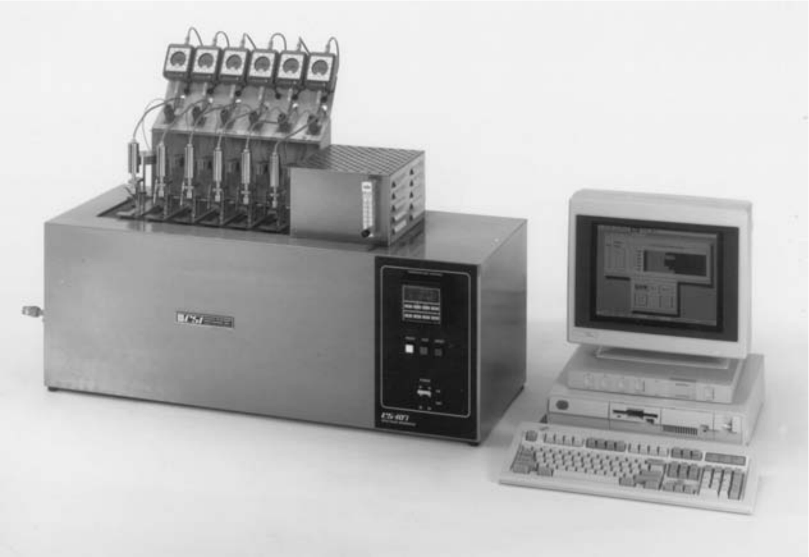


Fig. 15 DTUL/vicat tester. (Courtesy Atlas Electric Devices Company)

oil at which the bar has deflected 0.010 in. is recorded as the heat deflection temperature at the specified fiber stress.

Long-Term Effects

The long-term effects of elevated temperature on properties of plastics are extremely important, especially when one considers the fact that the majority of applications involving high heat are long-term applications. During long-term exposure to heat, plastic materials may encounter many physical and chemical changes. A plastic material that shows little or no effect at elevated temperature for a short time may show a drastic reduction in physical properties, a complete loss of rigidity, and severe thermal degradation when exposed to elevated temperature for a long time. Along with time and temperature, many other factors such as ozone, oxygen, sunlight, and pollution combine to accelerate the attack on plastics. At elevated temperatures, many plastics tend to lose important additives such as plasticizers and stabilizers, causing plastics to become brittle or soft and sticky.

Three basic tests have been developed and accepted by the plastics industry. If the application does not require the product to be exposed to elevated temperature for a long period under continuous load, a simple heat resistance test is adequate. The applications requiring the product to be under continuous significant load must be looked at from creep modulus and creep rupture strength test data. Another one of the most widely accepted methods of measuring maximum continuous use temperature has been developed by Underwriters Laboratories. The UL temperature index, established for a variety of plastic materials to be used in electrical applications, is the maximum temperature that the material may be subjected to without fear of premature thermal degradation.

Long-Term Heat Resistance Test (ASTM D794). The long-term heat resistance test was developed to determine the permanent effect of heat on any property by selection of an appropriate test method and specimen. In ASTM recommended practice, only the procedure for heat exposure is specified and not the test method or specimen.

Any specimen, including sheet, laminate, test bar, or molded part may be used. If a specific property, such as tensile strength loss is to be determined, a standard tensile test bar specimen and procedures must be used for comparison of test results before and after the test. The test requires the use of a mechanical convection oven with a specimen rack of suitable design to allow air circulation around the specimens. The test is carried out by simply placing the specimen in the oven at a desired exposure temperature for a predetermined length of time. The subsequent exposure to temperatures may be increased or decreased in steps of 25°C until a failure is observed. Failure due to heat is defined as a change in appearance, weight, dimension, or other properties that alter plastic material to a degree that it is no longer acceptable for the service in question. Failure may result from blistering, cracking, loss of plasticizer, or other volatile material that may cause embrittlement, shrinkage, or change in desirable electrical or mechanical properties.

Many factors that affect the reproducibility of the data. The degree of temperature control in the oven, the type of molding, cure, air velocity over the specimen, period of exposure, and humidity of the oven room are some of these factors. The amount and type of volatiles in the molded part or specimen may also affect the reproducibility.

UL Temperature Index. The increased use of plastic materials in electrical applications such as appliances, portable electrically operated tools and equipments, and enclosures has created a renewed interest in the ability of plastics to withstand mechanical abuse and high temperatures. A serious personal injury, electric shock, or fire may occur if the product does not perform its intended function. Underwriters Laboratories, an independent, not-for-profit organization concerned with consumer safety, has developed a temperature index to assist UL engineers in judging the acceptability of individual plastics in specific applications involving long-term exposure to elevated temperatures. The UL temperature index correlates numerically with the temperature rating or maximum temperature in degrees centigrade above which a material may degrade prematurely and therefore be unsafe.

Relative Thermal Indices. The relative thermal index of a polymeric material is an indication of the material's ability to retain a particular property (physical, electrical, etc.) when exposed to elevated temperatures for an extended period of time. It is a measure of the material's thermal endurance. For each material, a number of relative thermal indices can be established, each index related to a specific thickness of the material.

The relative index of a material is determined by comparing the thermal-aging characteristic of one material of proven field service at a particular temperature level with the thermal-aging characteristics of another material with no field service history. A great deal of consideration is given to the properties that are evaluated to determine relative thermal index. For the relative thermal index to be valid, the properties being stressed in the end product must be included in the thermal-aging program. If, for any reason, the specific property under stress in the end product is not part of the long-term aging program, the relative thermal index may not be applicable to the use of the material in that particular application.

Relative Thermal Index Based upon Historical Records. Through experience gained from testing a large volume of complete products and insulating systems over a long period, UL has established relative thermal indices on certain types of plastics. These fundamental temperature indices are applicable to each member of a generic material class. Table 2 lists the temperature indices based on past field test performance and chemical structure.

Relative Thermal Index Based upon Long-Term Thermal Aging. The long-term thermal-aging program consists of exposing polymeric materials to heat for a predetermined length of time and observing the effect of thermal degradation. To carry out the testing, an electrically heated mechanical convection oven is preferred, however, with some provisions, a noncirculating static oven may be employed. The specific properties to be evaluated in the thermal-aging program are to be as nearly as possible representative of the properties required in the end application.

Table 2 Relative Thermal Indices Based upon Past Field Test Performance and Chemical Structure[a]

Material	Generic Thermal Index (°C)
Nylon (type 6, 11, 6/6, and 6/10)[b]	65
Polycarbonate[b]	65
Molded phenolic[c,d]	150
Molded melamine[c,d]	130[e]
Molded melamine-phenolic[c,d]	130[e]
Fluorocarbin resins	
(1) Polytetrafluoroethylene	150
(2) Polychlorotrifluoroethylene	150
(3) Fluorinated ethylene propylene	150
Silicone rubber	105
Polyethylene terephthalate film	105
Urea formaldehyde	100
Molded alkyd[c,d]	130
Molded epoxy[c,d]	130
Molded diallyl phthalate[c,d]	130
Molded polyester[c,d] (thermosetting)	130

[a] From UL 746 B. Reprinted with permission from Underwriters Laboratories, Inc.

[b] Includes glass fiber-reinforced materials.

[c] Includes simultaneous heat and high-pressure matched metal die-molded compounds only. Excludes low-pressure or low-temperature curing processes such as open-mold (hand lay-up, spray-up, contact bag, filament winding), encapsulation, lamination, etc.

[d] Includes materials having filler systems of fibrous (other than synthetic organic) types but excludes fiber reinforcement systems using resins that are applied in liquid form.

[e] Compounds having a specific gravity of 1.55 or greater (including those having cellulosic filler material) are acceptable at temperatures not greater than 150°C (302°F).

The most common mechanical properties include tensile strength, flexural strength, and Izod impact strength. The electrical properties of concern are dielectric strength, surface or volume resistivity, arc resistance, and arc tracking. The test specimens are standard ASTM test bars, depending upon the type of test. The UL publication, "Polymeric Materials—Short-Term Property Evaluations, UL 746A," describes the specimen and test procedures to determine mechanical and electrical properties.

To determine the relative thermal index, a control material with a record of good field service at its rated temperature is selected. The control material of the same generic type and the same thickness as the candidate material is preferred. At least four different oven temperatures are selected. The highest temperature is selected so that it will take no more than 2 months to produce end of life of the material. The next two lower temperatures must produce the anticipated end of life of 3 and 6 months, respectively. The lowest temperature selected will take 9 to 12 months for the anticipated results.

The end of life of a material is based upon the assumption that at least a 2:1 factor of safety exists in the applicable physical and electrical property requirements. The end of life of a material is the time at each aging temperature, when a property value has decreased 50% of its unaged level. A 50% loss of property due to thermal degradation is not expected to result in premature, unsafe failure.

To avoid underrating the material's relative temperature index, UL publishes the ratings in three categories: applications involving electrical properties only, applications involving both electrical and mechanical properties, and applications involving both electrical and mechanical properties without impact resistance. UL publishes such data in its semiannual Plastics Recognized Component Directory. Since the long-term heat-aging resistance of plastics is dependent on the thickness of the part or test specimen, UL requires that the thermal index testing be carried out over a wide range of thicknesses. Finally, it is important to understand that the UL temperature index program recognizes that the upper temperature limits of plastics are dependent upon the stresses applied on the end product in use. Consequently, the temperature index of a particular plastic qualifies it only for those UL applications that UL has specifically approved.

2.2 Brittleness Temperature (ASTM D746, ISO 974)

At low temperatures, all plastics tend to become rigid and brittle. This happens mainly because, at lower temperatures, the mobility of polymer chains is greatly reduced. In the case of the amorphous polymers, brittle failure occurs at a temperature well below glass transition temperature. The polymer tends to get tougher as it reaches glass transition temperature. By contrast, in crystalline polymers the toughness is mainly dependent on the degree of crystallinity, which generally creates molecular inflexibility resulting in only moderate impact strength. The size of the crystalline structure formed also has significant effect on the impact strength of the polymer. The larger the crystalline structure, the lower the impact strength. The polymers exhibiting ductile failure generally show high plastic deformation characterized by material stretching and tearing before fracturing. Brittleness temperature is defined as the temperature at which plastics and elastomers exhibit brittle failure under impact conditions. Yet another way to define brittleness temperature is the temperature at which 50% of the specimens tested exhibit brittle failure under specified impact conditions.

3 ELECTRICAL PROPERTIES

The unbeatable combination of characteristics such as ease of fabrication, low cost, light weight, and excellent insulation properties have made plastics one of the most desirable materials for electrical applications. Although the majority of applications involving plastics are insulation related, plastics can be made to conduct electricity by simply modifying the base material with proper additives such as carbon black.

Until recently, plastics were considered a relatively weaker material in terms of load-bearing properties at elevated temperatures. Therefore, the use of plastics in electrical applications was limited to non-load-bearing, general-purpose applications. The advent of new high-performance engineering materials has altered the entire picture. Plastics are now specified in a majority of applications requiring resistance to extreme temperatures, chemicals, moisture, and stresses. The primary function of plastics in electrical applications has been that of an insulator. This insulator or dielectric separates two field-carrying conductors. Such a function can be served equally well by air or vacuum. However, neither air nor vacuum can provide any mechanical support to the conductors. Plastics not only act as effective insulators but also provide mechanical support for field-carrying conductors. For this very reason, the mechanical properties of plastic materials used as insulators become very important. Typical electrical applications of plastic material include plastic-coated wires, terminals, connectors, industrial and household plugs, switches, and printed circuit boards.

The key electrical properties of interest are dielectric strength, dielectric constant, dissipation factor, volume and surface resistivity, and arc resistance.

3.1 Dielectric Strength (ASTM D149, IEC 243-1)

The dielectric strength of an insulating material is defined as the maximum voltage required to produce a dielectric breakdown. Dielectric strength is expressed in volts per unit of thickness such as V/mil. All insulators allow a small amount of current to leak through or around themselves. Only a perfect insulator, if there is such an insulator in existence, can be completely free from small current leakage. The small leakage generates heat, providing an easier access to more current. The process slowly accelerates with time and the amount of voltage applied until a failure in terms of dielectric breakdown or what is known as puncture occurs. Obviously, dielectric strength, which indicates electrical strength of a material as an insulator, is a very important characteristic of an insulating material. The higher the dielectric strength, the better the quality of an insulator. Three basic procedures have been developed to determine dielectric strength of an insulator. Figure 16 schematically illustrates the basic setup for a dielectric strength test. A variable transformer and a pair of electrodes are normally employed. Specimens of any desirable thickness prepared from the material to be tested are used. Specimen thickness of $\frac{1}{16}$ in. is fairly common. The first procedure is known as the short-times method. In this method, the voltage is increased from zero to breakdown at a uniform rate. The rate of rise is gen-

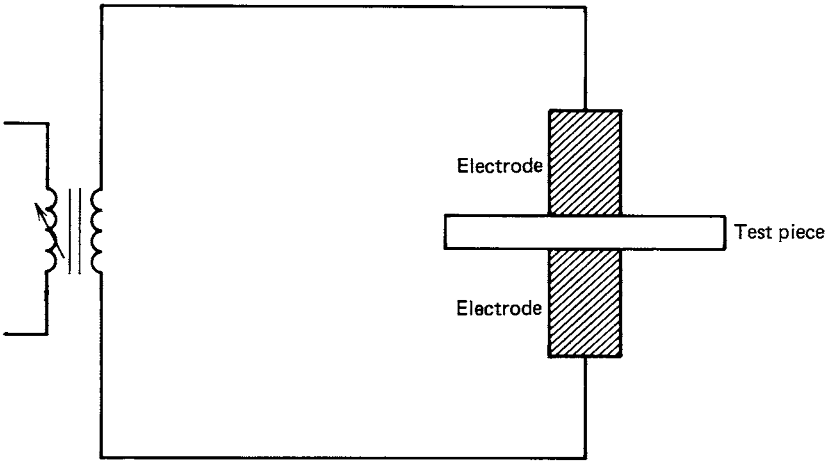


Fig. 16 Schematic of dielectric strength test.

erally 100, 500, 1000, or 3000 V/s until the failure occurs. The failure is made evident by actual rupture or decomposition of the specimen. Sometimes a circuit breaker or other similar device is employed to signal the voltage breakdown. This is not considered a positive indication of voltage breakdown since other factors such as flashover, leakage current, corona current, or equipment magnetizing current can influence such indicating devices.

The second method is known as the slow-rate-of-rise method. The test is carried out by applying the initial voltage approximately equal to 50% of the breakdown voltage as determined by the short-time test or as specified. Next, the voltage is increased at a uniform rate until the breakdown occurs.

The step-by-step test method requires applying initial voltage equal to 50% of the breakdown voltage as determined by the short-time test and then increasing the voltage in equal increments and held for specified time periods until the specimen breaks down. In almost all cases the dielectric strength values obtained by step-by-step method corresponds better with actual use conditions. However, the service failures are generally at voltage below the rated dielectric strength because of the time factor involved.

3.2 Dielectric Constant and Dissipation Factor (ASTM D150, IEC 250)

Dielectric Constant (Permittivity)

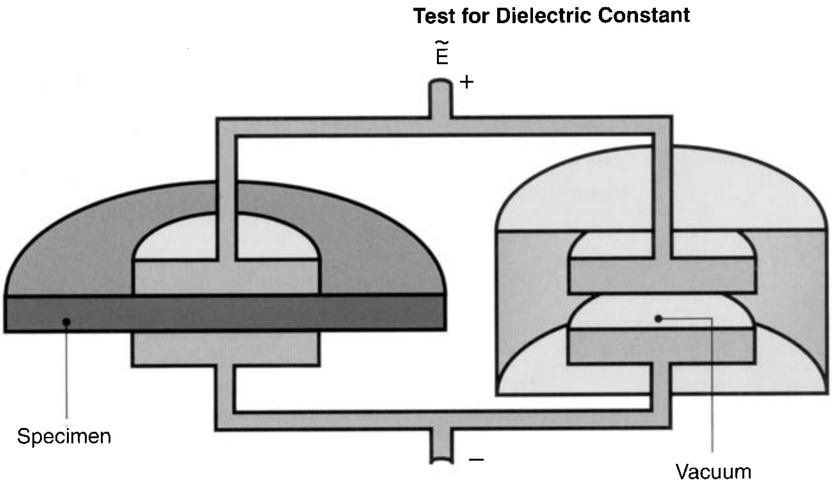
Dielectric constant of an insulating material is defined as the ratio of the charge stored in an insulating material placed between two metallic plates to the charge that can be stored when the insulating material is replaced by air (or vacuum). Defined another way, the dielectric constant is the ratio of the capacitance by two metallic plates with an insulator placed between them and the capacitance of the same plates with a vacuum between them. Simply stated, the dielectric constant indicates the ability of an insulator to store electrical energy. In many applications, insulating materials are required to perform as capacitors. Such applications are best served by plastic materials having a high dielectric constant. Materials with a high dielectric constant have also helped in reducing the physical size of the capacitors. Furthermore, the thinner the insulating material, the higher the capacitance. Because of this fact plastic foils are extensively used in applications requiring high capacitance.

One of the main function of an insulator is to insulate the current-carrying conductors from each other and from the ground. If the insulator is used strictly for this purpose, it is desirable to have the capacitance of the insulating material as small as possible. For these applications, one is looking for the materials with very low dielectric constant.

The dielectric constant test is fairly simple. The test specimen is placed between the two electrodes, as shown in Fig. 17, and the capacitance is measured. Next, the test specimen is replaced by air and once again the capacitance value is measured. The dielectric constant value is determined from the ratio of the two measurements. Dielectric constant values are affected by factors such as frequency, voltage, temperature, humidity, etc.

Dissipation Factor

In all electrical applications, it is desirable to keep the electrical losses to a minimum. Electrical losses indicate the inefficiency of an insulator. The dissi-



Dielectric constant is the ratio of the system capacitance with the plastic specimen as the dielectric to the capacitance with a vacuum as the dielectric.

Fig. 17 Schematic of dielectric constant test. (Courtesy Bayer Corporation)

pation factor is a measure of such electrical inefficiency of the insulating material. The dissipation factor indicates the amount of energy dissipated by the insulating material when the voltage is applied to the circuit. The dissipation factor is defined as the ratio of the conductance of a capacitor in which the material is the dielectric to its susceptance or the ratio of its parallel reactance to its parallel resistance. Most plastics have a relatively lower dissipation factor at room temperature. However, at high temperatures, the dissipation factor is quite high, resulting in greater overall inefficiency in electrical system. Loss factor, which is the product of dielectric constant and the dissipation factor, is a frequently used term since it relates to the total loss of power occurring in insulating materials.

3.3 Electrical Resistance Tests

As stated earlier, the primary function of an insulator is to insulate current-carrying conductors from each other as well as from ground and to provide mechanical support for components. Naturally, the most desirable characteristic of an insulator is its ability to resist the leakage of the electrical current. The higher the insulation resistance, the better the insulator. Failure to recognize the importance of insulation resistance values while designing products such as appliances and power tools could lead to fire, electrical shock, and personal injury.

Insulation resistance can be subdivided into:

1. Volume resistance
2. Surface resistance

Volume resistance is defined as the ratio of the direct voltage applied to two electrodes that are in contact with a specimen to that portion of the current between them that is distributed through the volume of the specimen. Or, simply stated, the volume resistance is the resistance to leakage through the body of the material. Volume resistance generally depends upon the material. The term most commonly used by designers is volume resistivity. It is defined as the ratio of the potential gradient parallel to the current in the material to the current density or, simply stated, the volume resistivity of a material is the electrical resistance between the opposite faces of a unit cube for a given material and at a given temperature.

High-volume resistivity materials are desirable in applications requiring superior insulating characteristics.

The surface resistance of a material is defined as the ratio of the direct voltage applied to the electrodes to that portion of the current between them that is primarily in a thin layer of moisture or other semiconducting material that may be deposited on the surface. Simply stated, surface resistance is the resistance to leakage along the surface of an insulator. The surface resistance of a material depends upon the quality and cleanliness of the surface of the product. A product with oil or dirt particles on it gives lower surface resistance values.

The test procedures to determine electrical resistance values are rather complex. ASTM D257 (IEC 93) describes the procedures as well as the complex electrodes and apparatus required to carry out the test in detail.

Temperature and humidity both seem to affect the insulation resistance appreciably. As a rule, the higher the temperature and humidity, the lower the insulation resistance of a material.

3.4 Arc Resistance (ASTM D495)

Arc resistance is the ability of a plastic material to resist the action of a high-voltage electrical arc, usually stated in terms of time required to form material electrically conductive. Failure is characterized by carbonization of the surface, tracking, localized heating to incandescence, or burning. In all applications in which conducting elements are brought into contact, arcing is inevitable. Switches, circuit breakers, and automotive distributor caps are a few good examples of applications where arcing is known to cause failure. Another term that is generally associated with arcing is tracking. Tracking is defined as a phenomenon where a high-voltage source current creates a leakage or fault path across the surface of an insulating material by slowly but steadily forming a carbonized path appearing as a thin, wiry line between the electrodes. Tracking is accelerated by the presence of surface contaminants such as dirt and oil and by the presence of moisture. Resistance to arcing or tracking depends upon the type of plastic materials such as phenolics that tend to carbonize easily and therefore have relatively poor arc resistance. Plastics such as alkyds, melamines, and fluorocarbons are excellent arc-resistant materials. The failures due to arcing are not always because of carbonization or tracking. Many plastics such as acrylics simply do not carbonize. However, they do form ignitable gases that cause the product to fail in a short time. Arc resistance of plastics can be improved substantially by the addition of fillers such as glass, mineral, wood flour, asbestos, and other inorganic fillers. Table 3 lists the arc resistance of some common plastic materials.

The determination of the arc resistance of plastics using a standard test method has always been a problem because of the numerous ways the test can

Table 3 Arc Resistance of Various Plastics

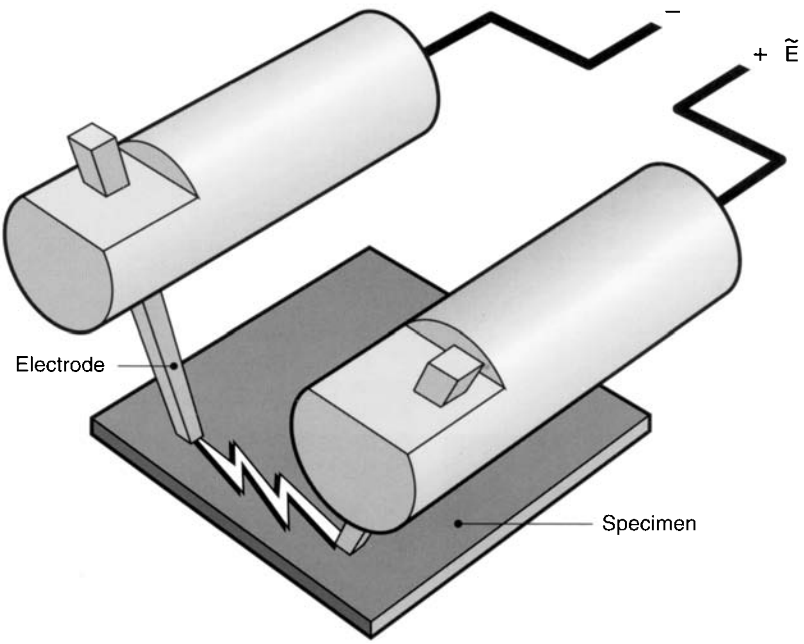
Plastics	Arc Resistance (sec)
Polycarbonate (10–40 percent glass filled)	5–120
Polycarbonate	10–120
Polystyrene (high impact)	20–100
ABS	50–85
Polystyrene (general purpose)	60–80
Rigid polyvinyl chloride	60–80
Polysulfone	75–190
Urea formaldehyde	80–150
Ionomer	90–140
SAN	100–150
Epoxy	120–150
Acetal (homopolymer)	130
Polyethylene (low-density)	135–160
Polypropylene	135–180
PTFE	>200
Acrylics	No track

be conducted. ASTM has developed four basic test methods to satisfy this concern. ASTM D495, which is high-voltage, low-current, dry arc resistance of solid electrical insulation, has been the most widely used and accepted. This test is only intended for the preliminary screening of materials, for detecting the effect of changes in formulations, and for quality control testing. Since the test is conducted under clean and dry laboratory conditions that are rarely encountered in practice, the prediction of materials from test results is next to impossible. Figure 18 illustrates a typical setup for an arc resistance test. The voltage is applied intermittantly and severity is increased in steps until the failure occurs. Arc resistance is measured in seconds to failure.

ASTM method D2132 outlines the procedure for determining dust and fog tracking and erosion resistance of electrical insulating materials. The test is carried out in a fog chamber with a standardized dust applied to the sample surface. Failure is characterized by the erosion of the specimen or tracking. ASTM D2302 also describes the test for differential wet tracking resistance of electrical insulating materials with controlled water-to-metal discharges. The inclined specimen is partially immersed in a water solution of ammonium chloride and a wetting agent. Failure is characterized by tracking. ASTM D2303 describes the test for liquid–contaminant inclined plane tracking and erosion of insulating materials. In this test, the specimen is inclined at 45°, and the electrolyte is discharged onto the surface at a controlled rate, increasing the voltage at the same time. The failure is marked by erosion and tracking.

Electromagnetic Interference/Radio Frequency Interface Shielding

The explosive growth of computers, telecommunication, and other electronic devices has created an urgent need for understanding and developing test and measurement techniques and standards for electromagnetic interference (EMI) and radio frequency interface (RFI) shielding. All electronic devices are required to have electromagnetic compatibility. It is a phenomenon caused by unrestricted



Arc resistance electrodes intermittently subject the specimen surface to a high-voltage arc until a conductive path is formed.

Fig. 18 Arc resistance test. (Courtesy Bayer Corporation)

electrical and magnetic energy that escapes from any electrical device and reaches a second unintended device. It is called EMI if the receiving device malfunctions as a result of this pollution. Any electrical signal falling between direct current (DC) electricity and the gamma-ray frequency region of the electromagnetic spectrum can be sources of EMI. For most practical purposes, the majority of EMI problems are limited to a part of spectrum from 1 kHz to 10 GHz. This portion of electromagnetic spectrum is known as the radio frequency interface (RFI) band and covers the radio and audio frequencies. EMI of some form is experienced by us everyday. Some of the most common sources of EMI are lightning, static electricity, television and radio receivers, electric motors, electric appliances, paging system, and radar transmitters. Some of the most common receptors of EMI are microprocessors, computers, hi-fi equipment, electronic measuring equipment, radio and television receivers, remote control units, aircraft navigation systems, and humans and animals.

The fact that electronic devices are both sources and receptors of EMI creates a twofold problem. Since electromagnetic radiation penetrating the device may cause electronic failure, manufacturers need to not only protect the operational integrity of their product but also must comply with the regulations aimed at reducing electromagnetic radiation emitted into the atmosphere.

The switch from metal to plastics as a housing material for electronic equipment has contributed to the EMI shielding issue. Plastics are insulators to electrical energy, so EMI waves pass freely through unshielded plastic without any impedance or resistance. Metals, being conductors, reflect or absorb electromagnetic energy, and unwanted electrical energy is easily grounded. Shielding, the term used to describe the effective ‘‘blocking’’ of EMI, is provided by a conductive barrier that harmlessly reflects or transmits electrical interference to ground.

EMI/RFI shielding of electronic devices is achieved in many different ways. Two of the most common ways to achieve shielding is either to coat the plastic housing with conductive material or to make the housing material itself conductive. The second method of using conductive plastics for shielding effectiveness is relatively new and for the most part in research and development stage with limited commercial applications. Coating methods on plastic parts are by far the most popular and the most varied shielding techniques. Basically, they all involve coating the finished plastic housing with a layer of conductive material to cerate a faraday cage (a concept that an enclosed conductive housing has a zero electrical field), resulting in a EMI shield. Electroless plating method is also gaining popularity as an alternate method to conductive coatings.

Shielding Effectiveness Measurement

Electromagnetic energy is most effectively measured in terms of attenuation. Attenuation is the amount by which the intensity of an electromagnetic signal is reduced by the introduction of a shielding medium. The attenuation is measured in decibels (dB) and is the ratio of the field strength without the shield to the field strength with the shield. Shielding effectiveness measured in decibels is a logarithmic scale, which means that an attenuation measurement of 50 dB is ten times more effective than a measurement of 40 dB.

Four basic techniques for measuring shielding effectiveness have been developed:

Open field testing
Shielded box
Coaxial transmission line
Shielded room

Conductivity Measurement

In production, conductivity testing becomes an important means for quality control of individual parts as well as for part-to-part testing of assembled enclosures. Surface resistivity, volume resistivity, and ohms-per-square measurement are common tests for both conductive coating and conductive plastics and used as a quick and practical quality check.

Surface Resistivity. Surface resistivity measures the resistance of a material to moving or distributing a charge over its surface. The lower the surface resistivity value, the easier a material will redistribute an electrical charge over its entire surface area. Surface resistivity test procedure is described in ASTM D257.

Surface resistivity meters that allow direct reading in ohms/square measurement are most commonly used. One of the meters uses a probe with an inner and an outer ring. A charge is applied to the inner ring, which travels over the surface of the material and then is recorded by the outer ring. In addition to these resistivity meters, manufactures often use a digital ohmmeter to measure the point-to-point resistance between two probes in contact with metallic coating. This method utilizes two spring-loaded probes that are placed at designated positions on the part or between two areas on mating parts. The conductivity between the two points is measured. This method is illustrated in Fig. 19.

Volume Resistivity. Volume resistivity measures the ability of a material to resist moving charge through its volume. The lower the volume resistivity of a material, the more conductive the material. This test procedure is also described in detail in ASTM D257.

Ohms-per-Square Testing. Ohms-per-square testing involves the placement of a probe on a surface of the part. This probe has two outside blocks of equal geometry and a space of equivalent dimension between the blocks. The probe is simply placed on the part and conductivity is measured. The test is extremely useful in quick checking the coated parts to verify minimum thickness of a conductive film. However, the test is not very effective for determining the overall conductivity of a coating across an actual part.

4 WEATHERING PROPERTIES

The increased outdoor use of plastics has created a need for a better understanding of the effect of the environment on plastic materials. The environmental factors have significant detrimental effects on appearance and properties. The severity of the damage depends largely on factors such as the nature of the environment, geographic location, type of polymeric material, and duration of exposure. The effect can be anywhere from a mere loss of color or a slight crazing and cracking to a complete breakdown of the polymer structure. Any attempt to design plastic parts without a clear understanding of the degradation mechanisms induced by the environment would result in a premature failure of the product. The major environmental factors that seriously affect plastics are:

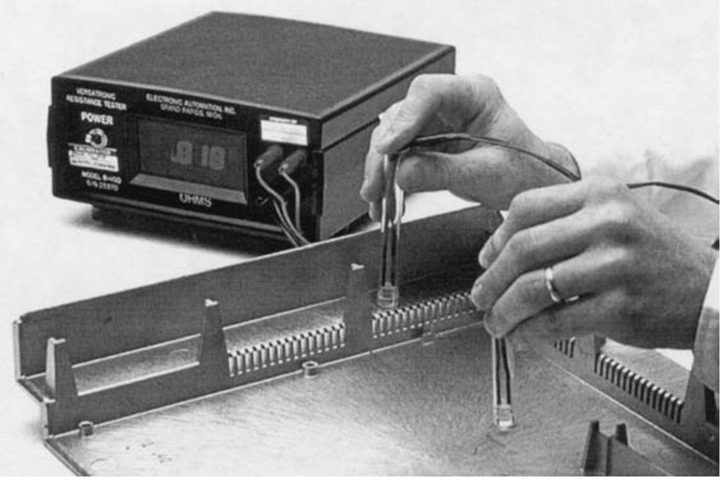


Fig. 19 Point-to-point resistance measurement. (Courtesy Enthone-EMI Corporation)

1. Solar radiations—ultraviolet (UV), infrared (IR), X-rays
2. Microorganisms, bacteria, fungus, mold
3. High humidity
4. Ozone, oxygen
5. Water: vapor, liquid, or solid
6. Thermal energy
7. Pollution: industrial chemicals

The combined effect of the factors mentioned above may be much more severe than the effect of any single factor, and the degradation processes are accelerated many times. Many test results do not include these synergistic effects, which almost always exist in real-life situations.

UV Radiation. All types of solar radiation have some sort of detrimental effect on plastics. Ultraviolet radiation is the most destructive of all radiations. The energy in ultraviolet radiation is sufficiently strong enough to break molecular bonds. This activity in the polymer brings about thermal oxidative degradation that results in embrittlement, discoloration, and an overall reduction in physical and electrical properties. Xenon arc lamps, fluorescent lighting, sun lamps, and other artificial sources also emit a similar type of harmful radiation. Other factors in the environment such as heat, humidity, and oxygen accelerate the UV degradation process.

One of the best methods of protecting plastics against UV radiation is to incorporate UV absorbers or UV stabilizers into the plastic materials. The UV absorbers provide preferential absorption to most of the incident UV light and are able to dissipate the absorbed energy harmlessly. Thus, the polymer is protected from harmful radiation at the cost of UV absorbers which are destroyed in the process with time. Several types of organic and inorganic UV absorbers, are developed for this purpose. Almost all inorganic pigments absorb UV radiation to a certain extent and provide some degree of protection. Perhaps the most effective pigments are certain types of carbon black that absorb over the entire range of UV and visible radiation and transform the energy into less harmful radiation.

UV stabilizers, unlike UV absorbers, inhibit the bond rupture by chemical means or dissipate the energy to lower levels that do not attack the bonds. The effectiveness of such additives when incorporated with the polymer can be determined by various test methods discussed in this chapter.

Microorganisms. Polymeric materials are generally not vulnerable to microbial attack under normal conditions. However, low-molecular-weight additives such as plasticizers, lubricants, stabilizers, and antioxidants may migrate to the surface of plastic components and encourage the growth of microorganisms. The detrimental effect can be seen readily through the loss of properties, change in aesthetic quality, loss of transmission (optical), and increase in brittleness. The rate of growth depends upon many factors, such as heat, light, and humidity. Preservatives, also known as fungicides or biocides, are added to plastic materials to prevent the growth of microorganisms. These additives are highly toxic to lower organisms but do not affect the higher organisms.

It is necessary to evaluate the effectiveness of various antimicrobial agents both on a laboratory scale as well as in actual outdoor exposure. Many such methods have been devised to perform these tests.

Oxygen, Moisture, Thermal Energy, and Other Environmental Factors. In addition to UV radiation and microorganisms, there are many other relevant factors that can add to polymer degradation. Even though most polymers react very slowly with oxygen, elevated temperatures and UV radiation can greatly promote the oxidation process. Water is considered relatively harmless; however, it can have at least three kinds of effects leading to early polymer degradation: a chemical effect, that is, the hydrolysis of unstable bonds; a physical effect, that is, destroying the bond between a polymer and filler resulting in chalking; the third is a photochemical effect. Thermal energy plays an indirect role in polymer degradation by accelerating hydrolyis, oxidation, and photochemical reactions induced by other factors. Many other factors such as ozone, atmospheric contaminants including dirt, soot, smog, sulfur dioxide, and other industrial chemicals, have a significant effect on polymers.

4.1 Accelerated Weathering Tests

Most data on the aging of plastics are acquired through accelerated tests and actual outdoor exposure. The latter being a time-consuming method, accelerated tests are often used to expedite screening the samples with various combinations of additive levels and ratios. A variety of light sources are used to simulate the natural sunlight. The artificial light sources include carbon arc lamps, xenon arc lamps, fluorescent sun lamps, and mercury lamps. These light sources, except fluorescent, are capable of generating a much higher intensity light than natural sunlight. Xenon arc lamps can be operated over a wide range from below peak sunlight to twice the sunlight levels. Quite often, a condensation apparatus is used to simulate the deterioration caused by sunlight and water as rain or dew. Modern instruments have direct specimen spray on front and/or back side of the specimen.

There are three major accelerated weathering tests:

1. Exposure to carbon arc lamps
2. Exposure to xenon arc lamps
3. Exposure to fluorescent UV lamps

The xenon arc, when properly filtered, most closely approximates the wavelength distribution of natural sunlight.

Exposure of Plastics to Fluorescent UV Lamps and Condensation (ASTM G53, ISO 4892)

This method is meant to simulate the deterioration caused by sunlight and dew by means of artificial ultraviolet light and condensation apparatus (Figs. 20 and 21). Solar radiation ranges from ultraviolet to infrared. Ultraviolet light of wavelengths between 290 and 350 nm is the most efficient portion of terrestrial sunlight that is damaging to plastics. In the natural sunlight spectrum, energy below 400 nm accounts for less than 6% of the total radiant energy. Since the

Fig. 20 UV light and condensation apparatus. (Courtesy Q-Panel Lab Products)

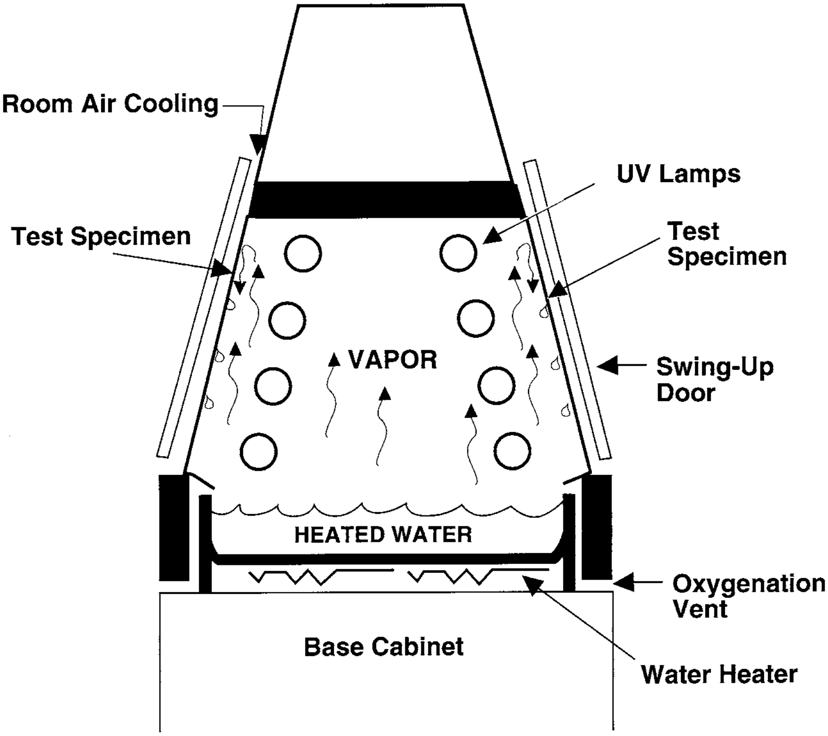


Fig. 21 Cross section of a UV light and condensation apparatus. (Courtesy Q-Panel Lab Products)

special fluorescent UV lamps radiate between 280 and 365 nm, they accelerate the degradation process considerably. In recent years, the UVA-340 lamps have increased in popularity because of the poor results of the conventional FS-40 lamps.

The test apparatus basically consists of a series of UV lamps, a heated water pan, and test specimen racks. The temperature and operating times are independently controlled both for UV and the condensation effect. The test specimens are mounted in specimen racks with the test surfaces facing the lamp. The test conditions are selected based on requirements and programmed into the unit. The specimens are removed for inspection at a predetermined time to examine color loss, crazing, chalking, and cracking.

Exposure of Plastics to Carbon Arc-Type Light and Water (ASTM D1499, ISO 4892)

This method is very useful in determining the resistance of plastic materials when exposed to radiation produced by carbon arc lamps. There are basically two different types of carbon arc lamps used as the source of radiation. The first type is an enclosed carbon arc lamp. The second type is known as an open-flame sunshine carbon arc. The enclosed carbon arc apparatus basically consists of either single or twin enclosed carbon arc lamps mounted in a chamber. The flame portion of the carbon arc lamp is surrounded by a bell-shaped borosilicate glass globe. The globe filters out UV radiation below 275 nm not found in direct sunlight and creates a semisealed atmosphere in which the arc burns more efficiently. The open-flame sunshine carbon arc lamp operates in a free flow of air. This lamp accommodates three upper and three lower electrodes that are consumed during 24–26 h of continuous operation. The lamp is typically surrounded by Corex glass filters that cut off light below 255 nm. The lamp is designed to run with filters in place; however, they can be removed to increase the available UV energy below 300 nm and thereby increase significantly the rate of photodegradation of UV-sensitive materials. The enclosed arc-type apparatus uses an open-ended drum-type cylinder, whereas the sunshine arc type uses a cylindrical framework. Both apparatuses revolve around centrally mounted arc lamps. The provision is also made to expose the specimen to water, which is sprayed through nozzles. The light-on, light-off, and water-spray cycles are independent of each other, and the apparatus can be programmed to operate with virtually any combination. A black panel temperature inside the test chamber can be monitored and controlled by a sensor mounted directly on the revolving specimen rack.

Exposure of Plastics to Xenon Arc-Type Light and Water (ASTM D2565, G26, ISO 4892)

A filtered xenon arc-type light source is one of the most popular indoor exposure tests since it exhibits a spectral energy distribution of sunlight at the surface of the earth. The xenon arc lamp consists of a burner tube and a light filter system with interchangeable glass filters used in combination to provide a spectral distribution that approximates natural sunlight exposure conditions. The apparatus typically has a built-in recirculating system that recirculates distilled or deionized water through the lamp. The water cools the xenon burner and filters out long wavelength infrared energy. For air-cooled lamps, this is accomplished by the use of optical filters.

Two basic procedures are recommended. Procedure A is a normal operating procedure for comparative evaluation within a series exposed simultaneously in one instrument. Procedure B is used for comparing results among instruments. Both procedures are described in detail in the ASTM *Standards Manual*. In either case, it is highly recommended that a controlled irradiance exposure system be used. This is best accomplished through the use of a continuously controlled monitor that can automatically maintain uniform intensity at preselected wavelengths or wavelength range, when broadband control is being used.

Interpretations and Limitations of Accelerated Weathering Test Results

There is a general agreement among the researchers, manufacturers, and users that the data from accelerated weathering tests cannot be correlated exactly with the results of natural weathering. However, accurate ranking of the weatherability of most material is possible by using improved test methods and sophisticated equipment. Accelerated weathering tests were devised to study the effect of actual outdoor weather in a relatively short time period. These tests often produce misleading results that are difficult to interpret or correlate with the results of actual outdoor exposure. The reason for such a contradiction is that in many laboratory exposures, the wavelengths of lights are distributed differently than in normal sunlight, possibly producing effects different from those produced by outdoor weathering. All plastics seem to be especially sensitive to wavelengths in the ultraviolet region. If the accelerated device has unusually strong emission at the wavelength of sensitivity of a particular polymer, the degree of acceleration is disproportionately high compared to outdoor exposure. The temperature of the exposure device also greatly influences the rate of degradation of a polymer. The higher temperature may cause oxidation and the migration of additives, which, in turn, affects the rate of degradation. One of the limitations of accelerated weathering devices is their inability to simulate the adverse effect of most industrial environments and many other factors present in the atmosphere and their synergistic effect on polymers. Some of the newly developed gas exposure cabinets have partially overcome these limitations. These units are capable of generating ozone, sulfur dioxide, and oxides of nitrogen under controlled conditions of temperature and humidity.

Improved ultraviolet sources and more knowledge of how to simulate natural wetness now make it possible to achieve reliable accelerated weathering results. Before drawing any final conclusions concerning the ability of a polymer to withstand outdoor environment based on accelerated weathering tests, one must conduct actual outdoor exposure tests for a reasonable length of time.

4.2 Outdoor Weathering of Plastics (ASTM D1435)

The test is devised to evaluate the stability of plastic materials exposed outdoors to varied influences that comprise weather exposure conditions that are complex and changeable. Important factors are climate, time of year, and the presence of industrial atmosphere. It is recommended that repeated exposure testing at different seasons and over a period of more than one year be conducted to confirm exposure at any one location. Since weathering is a comparative test, control samples are always utilized and retained at standard conditions of temperature and humidity. The control samples must also be covered with inert wrapping to exclude light exposure during the aging period. However, dark storage does not ensure stability.

Test sites are selected to represent various conditions under which the plastic product will be used. Arizona is often selected for intense sunlight, wide temperature cycle, and low humidity. Florida, on the other hand, provides high humidity, intense sunlight, and relatively high temperatures.

Exposure test specimens of suitable shape or size are mounted in a holder directly applied to the racks. Racks are positioned at a 45° angle and facing the equator. Many other variations in the position of the racks are also employed, depending upon the requirements.

The specimens are removed from the racks after a specified amount of time and subjected to various tests such as appearance evaluation, electrical tests, and mechanical tests. The results are compared with the test results from testing control specimens. Typical aluminum exposure racks are shown in Fig. 22.

Outdoor Accelerated Weathering

To accelerate the outdoor weathering, a reliable method for predicting long-term durability in a shorter time frame had to be developed. Outdoor accelerated test methods were developed some 30 years ago by DSET Laboratories. The method employs Fresnel-reflecting solar concentrators that use 10 flat mirrors to uniformly focus natural sunlight onto specimens mounted in the target plane. High-quality, first-surface mirrors provide an intensity of approximately eight suns with spectral balance of natural sunlight in terms of ultraviolet integrity. The test method provides an excellent spectral match to sunlight, correlating well to subtropical conditions such as southern Florida as well as an arid desert environment such as Arizona.

Fig. 22 Typical aluminum exposure racks. (Courtesy Atlas Electric Devices Company)

Fig. 23 Accelerated outdoor weathering testers. (Courtesy Atlas Electric Devices Company)

The test apparatus is a follow-the-sun rack with mirrors positioned as tangents to an imaginary parabolic trough. The axis is oriented in a north–south direction, with the north elevation having the capability for periodic altitude adjustment.

The target board, located at the focal line of the mirrors, lies under a wind tunnel along which cooling air is deflected across the specimens. A nozzle assembly is employed to spray the specimens with deionized water in accordance with established schedules. Night-time spray cycles can be used to keep specimens moist during the nontracking portion of the test. The entire 3-year real-time Florida exposure test can be carried out in just 6 months depending on the program start date. The test is widely used in automotive, agriculture, building, textile, and packaging industries. Figure 23 illustrates outdoor accelerated weathering test apparatus.

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CHAPTER 21

CHARACTERIZATION AND IDENTIFICATION OF PLASTICS

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1 MATERIAL CHARACTERIZATION TESTS

The plastics industry has grown phenomenally in the past two decades. Plastic material consumption has quadrupled. Today there are countless numbers of processors consuming in excess of 100 million pounds of material every year. An increasing number of processors are looking into various techniques for characterizing the incoming plastic resin to guard against batch-to-batch variations. Such variation in the properties and the processability of the polymer have been very costly.

To understand the phenomenon of batch-to-batch variation, one must understand the polymerization process. The polymerization mechanism can often be very complex. Basically, it involves adding a monomer or a mixture of monomers into a reaction kettle along with several other ingredients such as a catalyst, an initiator, and water, depending on the type of polymerization process. The contents are agitated at a specific speed. The time, temperature, and pressure are carefully controlled. During this process, the monomer is converted into a polymer. The properties of the final product depend on several factors such as the monomer-to-water ratio, if water is present, the degree of agitation, the removal of exothermic heat generated during the polymerization, and the ability of the polymer to dissolve in monomer. This is further complicated by the fact that different polymerization techniques are used to form the same chemical type of polymer. Since it is difficult to manufacture a monodispersed polymer (a polymer

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Handbook of Materials Selection. Edited by Myer Kutz

in which all molecules have the same size) commercially, we are forced to live with variations in the size and weight of molecules in a polymer. Such variations in the size and the weight of the polymer molecules are also extremely difficult to control. The relative proportions of molecules of different weights within a polymer comprise its molecular weight distribution. Depending on the range of distribution (narrow or broad), the processibility and the properties of a polymer vary significantly. This basic nature of the polymerization process creates a need for material characterization tests that can be used as an insurance against variations in the characteristics of the polymer.

Table 1 lists various applications using rheometry and thermal analysis techniques.

There are numerous ways of characterizing a polymer. Some are very basic and simple, others are more sophisticated and complex. The five most common and widely accepted tests are:

Melt index (melt flowrate) test, rheological tests
Viscosity tests
Gel permeation chromatography
Thermal analysis [thermogravimetric analysis (TGA), thermomechanical analysis (TMA), and differential scanning calorimetry (DSC)]
Spectroscopy

1.1 Melt Index Test (ASTM D1238, ISO 1133)

The melt index, also known as melt flow rate (MFR), test measures the rate of extrusion of a thermoplastic material through an orifice of specific length and diameter under prescribed conditions of temperature and pressure. This test is primarily used as a means of measuring the uniformity of the flowrate of the material. The reported melt index values help to distinguish between the different grades of a polymer. A high-molecular-weight material is more resistant to flow than a low-molecular-weight material. However, the data obtained from this test does not necessarily correlate with the processibility of the polymer. This is due to the fact that plastic materials are seldom manufactured without incorporating additives that affect the processing characteristics of a material such as stability and flowability. The effect of these additives is not readily observed via the melt index test. The rheological characteristics of polymer melts depend on a number of variables. Since the values of these variables may differ substantially from those in large-scale processes, the test results may not correlate directly with processing behavior.

The melt index apparatus (Fig. 1) is preheated to a specified temperature. The material is loaded into the cylinder from the top and a specified weight is placed on a piston. The material is allowed to flow through the die. The initial extrudate is discarded since it may contain some air bubbles and contaminants. Depending on the material or its flowrate, cuts for the test are taken at different time intervals. The extrudate is weighed and melt index values are calculated in g/10 min. A commercial melt indexer is shown in Fig. 2.

The melt index values obtained from the test can be interpreted in several different ways. First of all, a slight variation (up to 10% in the case of polyeth-

Table 1 Characterization through Rheology and Thermal Analysis: Applications + Instruments = Solutions

	Rheometry			Thermal Analysis				
Application	Torque Rheometer	Rotational Rheometer	Capillary Rheometer	DMA	DSC	TG/DTA	TMA	DES
Bubble stability-blown film	✓	✓						
Compositional analysis					✓	✓		
Compounding	✓		✓					
Creep and stress relaxation		✓		✓			✓	
Curing studies	✓	✓		✓	✓			
Degree of cross-linking				✓	✓			
Dielectric properties								✓
Die swell	✓		✓					
Electrorheology		✓						
Elongational viscosity			✓					
Gamma, beta transitions				✓			✓	
Gel point		✓	✓				✓	
Glass transition				✓	✓	✓	✓	✓
Haze and gloss of films	✓	✓						
Heat capacities					✓			
Impact resistance		✓		✓			✓	
Lifetime predictions		✓		✓				
Measure degree of dispersion	✓	✓		✓	✓			
Measure rheological properties	✓	✓	✓	✓			✓	
Melt strength		✓	✓					
Melting temperatures	✓			✓	✓	✓	✓	
Molecular weight	✓	✓	✓	✓				
Molecular weight distribution	✓	✓	✓	✓				
Oxidative stability	✓				✓			
Processibility	✓	✓	✓		✓			
Sample preparation	✓							
Shear viscosity	✓	✓	✓					
Shrinkage forces							✓	
Tack and peel of pressure-sensitive additives		✓		✓			✓	
Thermal stability	✓	✓	✓	✓	✓	✓		
Viscoelastic properties		✓		✓			✓	

Table 1 (*Continued*)

Information	Process[a]	Data obtainable[b]
Composition		
Resins, polymers modifiers	DSC	Types present, plus ratio if copolymer, blend or mixture
	GC	Polymers, oligomers, and residual monomer; amounts present
Additives	DSC	Deduce presence and amount from thermal effects (stabilizers, antioxidants, blowing agents, plasticizers, etc.)
	GC	Detect any organic additive (by MW)
Reinforcement, fillers	TG	Amount (from weight of ash)
Moisture, volatiles	TG	Amounts (from weight loss)
	DSC	Amounts (if sufficient heat absorbed)
Regrind level	DSC	Deduce amount from melting-point shift
	GC	Deduce amount from shift in MW distribution
Processability		
Melting behavior	DSC	Melt point and range (each resin, if blend); melt-energy requirements
Flow characteristics	GC	Deduce from balance of high and low MW polymer in MW distribution
Physical, Mechanical		
Glass transition temperature	DSC	Shown by step-up in energy absorption as resin heats (marginal for some semicrystalline resins)
	TM	Detected by sample's expansion
Crystallinity	DSC	Calculate percentage from heat of fusion during melting; also can find time–temperature conditions for desired percent crystallinity
Temsile, flexibility, impact	GC	Deduce from balance of high and low MW polymer in MW distribution
	DSC	Deduce from overall composition
Cure Characteristics, Thermosets		
Reactive systems	DSC	Shows gel time, cure temperature, and cure time
Prepreg	DSC	Measures unreacted resin in B-stage prepreg or after cure (by volatiles)
	GC	As above, but checks by MW analysis of composite residue

[a] DSC = differential scanning calorimeter; GC = gel chromatography; TM = thermomechanical analysis.

[b] These techniques do not specifically identify materials. Materials can be identified by matching the instrument's test curves with those of known polymers or compounds. Otherwise other analytical procedures must be used. MW = molecular weight.

(Courtesy Haake-USA)

ylene) in the melt index value should not be interpreted as indicating a suspect material. The material supplier should be consulted to determine the expected reproducibility for a particular grade of plastic material. A significantly different melt index value than the control standard may indicate several different things. The material may be of a different grade with a different flow characteristic. It also means that the average molecular weight or the molecular weight distribution of the material is different than the control standard and may have different properties.

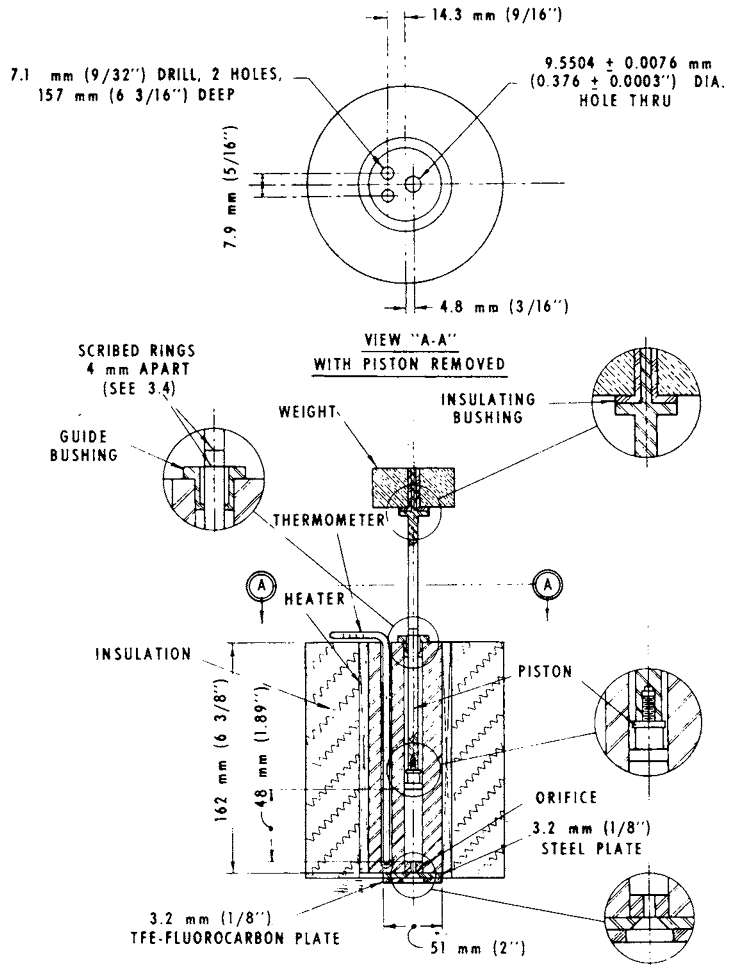


Fig. 1 Schematic of melt indexer. (Reprinted by permission of ASTM.)

Melt index is an inverse measure of molecular weight. Since flow characteristics are inversely proportional to the molecular weight, a low-molecular-weight polymer will have a high melt index value and vice versa.

1.2 Rheology

Rheology is defined as study of flow. Viscosity is the measure of resistance to flow due to internal friction when one layer of fluid is caused to move in relationship to another layer. The greater the friction, the greater the amount of force required to cause this movement, which is called *shear*. Shearing occurs whenever the fluid is physically moved or disturbed as in pouring, mixing, and spraying. Highly viscous fluids, therefore, require more force to move then less viscous materials. The velocity gradient is a measure of the speed at which the intermediate layers move with respect to each other. It describes the shearing the liquid experiences and is thus called *shear rate*. Shear stress is the stress

Fig. 2 Melt indexer. (Courtesy Dynisco-Kayeness Polymer Test Systems)

developing in the polymer melt when the layers in a cross section are gliding along each other. When the ratio of shearing stress to the rate of shear is constant, as in case of water, the fluid is called a *Newtonian fluid*. In the case of non-Newtonian fluids, the ratio varies with shearing stress or stated another way, when shear rate is varied, the shear stress does not vary in the same proportion. The viscosity of such fluids will change with the change in shear rate. All polymeric materials exhibit non-Newtonian flow behavior. This information concerning the flow of polymeric material is very useful in identification and characterization of the polymers. Rheological measurements are very useful in the determination of molecular, elastic, physical properties of polymers, simulation, determination of large-scale processing conditions, and basic research and development. Instruments developed for measuring the flow properties of polymers are known as rheometers. Many different types of rheometers have been developed. Data analysis and interpretation has been simplified with newly developed Windows-based software.

The following describes three major types of rheometers, most commonly used in the industry.

Torque Rheometer

Torque rheometry is used extensively for measuring the rheological properties as well as determining melt flow values, thermal stability of polymers, degradation time, and characterization of different formulations.

Rotational Rheometers

Many types of rotational rheometers have been developed over the years. Cone and plate, couette, torsional, and disc are most common.

The Spindle Viscometer (ASTM D2393) is one of the simplest instruments to use of all the rheometers used today. It measures torque required to rotate an immersed element (the spindle) in a fluid. The spindle is driven by a synchronous motor through a calibrated spring; the deflection of the spring is indicated by a pointer and a dial or a digital display. By utilizing a multiple speed transmission and interchangeable spindles, a variety of viscosity ranges can be measured. The sample is preconditioned by placing it in a constant temperature bath at the specified test temperature. The proper sized spindle is allowed to rotate in the sample for 2 min. The instrument is stopped through the use of a clutch, and the reading is taken from the dial or read directly from the digital display. The test is repeated until a constant reading is obtained. Viscosity is calculated using the factors provided with the viscometer. Data can be formatted to a printer or can be linked to a computer using a special software package. The newer model viscometers are programmable and have the capability of measuring and displaying viscosity and temperature simultaneously. Figure 3 shows a digital programmable viscometer.

Cone-and-plate viscometer is ideal for determining absolute viscosity of fluids in a small sample volume. The commercially available cone-and-plate viscometer (Wells-Brookfield), a precise torque measuring system, which consists of a calibrated beryllium–copper spring connecting the drive mechanism to a rotating cone, senses the resistance to rotation caused by a presence of sample fluid between the cone and a stationary flat plate. The resistance to the rotation of the cone produces a torque that is proportional to the shear stress in the fluid. The amount of torque is indicated on a dial or displayed digitally.

Capillary Rheometer

Quite often, the melt index test results simply do not provide sufficient information. This is because the melt index is a single-point test. The flowrates are measured at a single shear stress and shear rate performed at one set of temperatures and geometric conditions. Furthermore, since the melt index measurement takes account of the behavior of the polymer at only one point, it is quite possible for two materials with the same melt index values to behave completely different at shear stresses that are different from the ones used during the melt index measurements. The capillary rheometer measures apparent viscosity or melt index over an entire range of shear stresses and shear rates encountered in compression molding, calendering, extrusion, injection molding, and other

Fig. 3 Digital programmable viscometer. (Courtesy Brookfield Engineering Laboratories)

polymer melt processing operations. A rheometer is a precision instrument that provides the accuracy and reproducibility necessary for polymer characterization test.

A quality control test to qualify the incoming material is quick, simple, and accurate. The capillary rheometer can be used to assure that a material complies with the flow tolerances set for that grade of material. A quality control graph of a polymer flow curve can be generated with upper and lower critical viscosity limits over a wide range of shear rates by running multiple tests on a standard material. The thermal stability of a polymer and resulting processing limitations are of extreme importance to a processor. Capillary rheometer can accurately predict the thermal stability of the polymer. Materials such as polyvinyl chloride (PVC) can begin to cross-link and exhibit a dramatic increase in viscosity if left in the process too long. This can lead to rejected or failure-prone parts and wasted time and materials. The capillary rheometer allows the residence time capabilities of materials to be studied at wide range of processing temperatures and shear rates so the optimal processing temperature can be determined. The data generated from capillary rheometer measurements can be used to study the processibility of regrind material as well as process optimization. The capillary rheometer consists of an electrically heated cylinder, a pressure ram, temperature controllers, timers, and interchangeable capillaries. The plunger can be moved at a constant velocity, which translates to a constant shear rate. The force to move the plunger at this speed is recorded, which determines the shear stress. Alternately, a weight or constant pressure can be applied to the plunger, which generates a constant shear stress, and the velocity of the plunger can be determined by cutting and weighing the output. Shear rate can be calculated by knowing the melt density.

The sample material is placed in the barrel of the extrusion assembly, brought to temperature, and forced out through a capillary. The force required to move the plunger at each speed is detected by a load cell. Shear stress, shear rate, and apparent melt viscosity are calculated. Shear stress versus shear rate and apparent viscosity versus apparent shear rate curves are plotted.

1.3 Viscosity Tests

Viscosity is defined as the property of resistance to flow exhibited within the body of a material expressed in terms of a relationship between the applied shearing stress and the resulting rate of strain in shear. In the case of ideal or Newtonian viscosity, the ratio of shear stress to shear rate is constant. Plastics typically exhibit non-Newtonian behavior, which means that the ratio varies with the shearing stress. There are two different aspects of viscosity. Dynamic or absolute viscosity, best determined in a rotational type of viscometer with a small gap clearance, is independent of the density or specific gravity of the liquid sample and is measured in poises (P) and centipoises (cP). Kinematic viscosity, usually determined in some form of efflux viscometer equipped with a capillary bore or small orifice that drains by gravity, is strongly dependent on density or specific gravity of the liquid and is measured in stokes (S) and centistokes (cS). The relationship between two types of viscosity is

$$\text{stokes} \times \text{specific gravity} = \text{poises}$$

The measurement and control of rheological properties are usually performed with simple devices called "viscometers" or "viscosimeters," which do not measure true viscosities of either the dynamic or kinematic type but make relative flow comparisons. Viscosity of plastic materials is measured in three basic ways, employing principles involving liquid deformation due to various forces.

1. Downward rate of gravity flow through capillary bores and small orifices (capillary viscometers).
2. Upward speed of a trapped air bubble.
3. Torque developed by the liquid drag between moving and stationary surfaces.

The first method is basically used for thermoplastic materials. The latter two are more commonly used for thermosetting materials, plastisols, and organosols and are discussed in Section 1.2.

1.4 Gel Permeation Chromatography

Quite often traditionally used tests such as melt index or viscosity tests do not provide enough information about the processibility of the polymer. Such tests only measure an average value and tell us nothing about the distribution that makes up the average. Take, for instance, average daily temperatures reported for a particular city. These reports can be very misleading since they lack more useful information about extreme high and extreme low temperatures. In a very similar manner, the melt index and viscosity tests relate very well to the average molecular weight of the polymer but fail to provide the necessary information about the molecular weight distribution of the polymer. Two batches of resin may have the same melt index, which simply indicates that their viscosity average molecular weights are similar. Their molecular weight distributions, the number of molecules of various molecular weights that make up their averages, can be significantly different. If an excessively high-molecular-weight fraction is present, the material may be hard and brittle. Conversely, if an excessive amount of low-molecular-weight fraction is present, the material may be soft or sticky. When evaluating the nature of the incoming plastics material is extremely important to a processor, he must look for a more reliable technique that will provide the necessary information, to qualify the material, such as the measurement of molecular weight distribution. The molecular weight distribution is the single most fundamental property of the polymer. The molecular weight distribution not only provides basic information regarding the processibility of the polymer but also gives valuable information for predicting its mechanical properties.

Gel permeation chromatography (GPC) is the method of choice for determining the molecular weight distribution of a polymer. This technique has gained wide acceptance among the plastic material manufacturers and the processors because of its relatively low cost, simplicity, and its ability to provide accurate, reliable information in a very short time. GPC reveals the molecular weight distribution of a polymer compound. It detects not only the resin-based molecules such as polymer, oligomer, and monomer but most of the additives used in plastic compounds and even low-level impurities. The molecular weight distribution curve is plotted for a well-characterized standard material, and the profile of the curve is compared with the test sample. In this manner, batch-to-batch uniformity can be checked quickly as a means of quality assurance.

The separation of polymer molecules by GPC is based upon the differences in their "effective size" in solution. (Effective size is closely related to molecular weight.) Separation is accomplished by injecting the polymer solution into a continuously flowing stream of solvent that passes through highly porous, tiny, rigid gel particles closely packed together in a tube. The pore size of the gel particles may vary from small to very large. As the solution flows through the gel particles, molecules with small effective size (low molecular weights) will penetrate more pores than molecules with larger effective sizes and, therefore, take longer to emerge than the larger molecules. If the gel covers the right range of molecular sizes, the result will be a size separation with the largest molecules exiting the gel-packed tubes (columns) first.

A test sample is prepared by dissolving a small amount of polymer in the solvent and filtering the solution to remove the undissolved impurities. The next step is to select the proper size columns, connect them, set the sensitivity setting on the detectors, and allow the instrument to equilibrate. A trial analysis is done by injecting the polymer solution into the instrument. The chromatogram is carefully analyzed. If the chromatogram shows all the desired information, the final analysis is carried out. If not, the operating parameters, such as column size, flowrate, and number of columns are optimized. The trial step is repeated before proceeding to the final analysis.

During the final analysis, as the sample flows through the column, the molecules are separated according to size by a simple mechanical effect. Because of their smaller size, the smaller molecules enter into the gel pores more readily and, therefore, take extra time to reach the bottom of the column. The various molecular weight species are separated by the difference in travel time through the column and pass through a detector in descending order of size. The detector measures the concentration of each molecular size and plots the molecular weight distribution of the sample on a strip chart. Commercially available GPC equipment is shown in Fig. 4.

A GPC curve can reveal a great deal of information. The regions of the chromatogram can generally be easily and quickly correlated with the molecular weights of the components of the polymer. The most direct—and sometimes the most informative—use of the GPC curve is in the comparison of different materials by overlaying their chromatograms. Differences in molecular weight distributions, peak shapes, shifts, and tailing are readily observable. Comparisons of additives and other lower molecular weight species are straightforward.

Frequently, a master chromatogram representing the acceptable range of GPC profiles is established with samples from "good" batches. All subsequent batches are then chromatographed and compared with the master curve as a rapid quality control method. A number of correlations between the GPC curve and the physical and processing behaviors of a polymer have been developed.

Fig. 4 GPC equipment. (Courtesy Waters Corporation)

1.5 Thermal Analysis Techniques

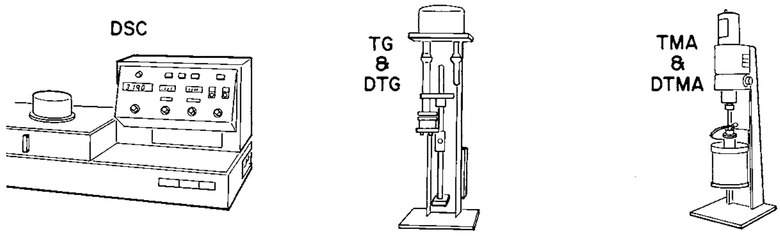
Thermal analysis (TA) consists of a family of analytical techniques in which a property of the sample is monitored against time or temperature while the temperature of the sample is programmed. Properties include weight, dimension, energy take-up, differential temperature, dielectric constant, mechanical modulus evolved gases, and other, less common attributes. The application of thermal analysis is widespread within the polymer and elastomer industries. Thermal analyzers are used in industry to qualify a material for fitness of use and to troubleshoot processing problems. Thermal analysis consists of three primary techniques that may be used individually or in combination:

1. Differential scanning calorimetry (DSC)
2. Thermogravimetric analysis (TGA)
3. Thermomechanical analysis (TMA)

The maximum benefit of thermal analysis can be gained by using combination of all three techniques to characterize a polymer. Figure 5 shows the three most common techniques and the test methods for each. The following sections describe the more common techniques in terms of analytical capability and standard methodology.

Differential Scanning Calorimetry (ASTM D3417, ASTM D3418)

Differential scanning calorimetry (DSC), the most widely used thermal analysis technique, is a technique in which the heat flowrate to the sample (differential power) is measured while the temperature of the sample, in a specified atmosphere, is programmed. Because all materials have a finite heat capacity, heating or cooling a sample specimen results in a flow or heat in or out of the sample.



MEASURE HEAT FLOW

- MELTING PROFILE OR T_g
- PROCESSING ENERGY, C_p
- EXPLOSIVE HAZARD
- % CRYSTALINITY
- CURING PROFILE
- BLEND, COPOLYMER ANALYSIS
- ADDITIVE ANALYSIS (MOLD-RELEASE, ANTISTAT, IMPACT AND VISCOSITY MODIFIERS)

MEASURES WEIGHT CHANGES

- % VOLATILES - H_2O, SOLVENT, MONOMER
- % PLASTICIZER, OIL-EXTENDER
- % CARBON BLACK
- % CARBONATE
- % INERT MATERIAL
- DEGRADATION PROFILES
- THERMAL, OXIDATIVE STABILITY

MEASURES DIMENSIONAL CHANGES

- THERMAL EXPANSION
- SOFTENING POINT
- HEAT DEFLECTION TEMP.
- MODULUS, CREEP
- THERMAL REORIENTATION
- SHRINKAGE FROM MOLD

Fig. 5 Thermal analysis of plastics. (Courtesy Perkin-Elmer Corporation)

The two most common types of commercial DSC measuring cells are shown in Fig. 6. The heat flux DSC employs a disk containing sample and reference positions that are heated by a common furnace. The differential heat flow to the sample is proportional to the temperature difference that develops between sample and reference junctions of a thermocouple. The power compensations approach controls a temperature enclosure around the sample and reference individually. Through amplified feedback from platinum resistance thermometers, it records the differential energy flow necessary to maintain the sample on the specified temperature program. With either approach the output is heat flow, normally expressed in milliwatts, watts/gram (normalized) or watts/gram-degree (specific heat units).

The test procedure is simple. A small quantity of sample, usually 5–10 mg, is weighed out into a inert capsule (usually made of aluminum). The encapsulated sample is placed in the DSC sample holder or onto the sample platform of a DSC cell disk. In the attached control module or computer the operator selects a temperature range and heating rate or perhaps a more complex temperature program. The test is started. (A hypothetical DSC curve showing both endothermic and exothermic changes in a polymer is shown in Fig. 7.) Initially, constant energy input is required to heat the sample at a constant rate. This establishes a baseline. At a transition point, the sample requires either more or less energy depending on whether the change is endothermic or exothermic. For example, when the glass transition point is reached, the heat capacity increases. The midpoint is taken as the glass transition temperature, T_g. Adding plasticizers to a formulation lowers T_g. When a polymer reaches the melting point, it requires more energy (endothermic) to melt the crystalline structure. The area of the peak in units of energy is the enthalpy of fusion, the heat of melting. The temperature dependence of the peak and its shape give information about degree of crystallinity, the molecular weight distribution, degree of branching, copolymer blend

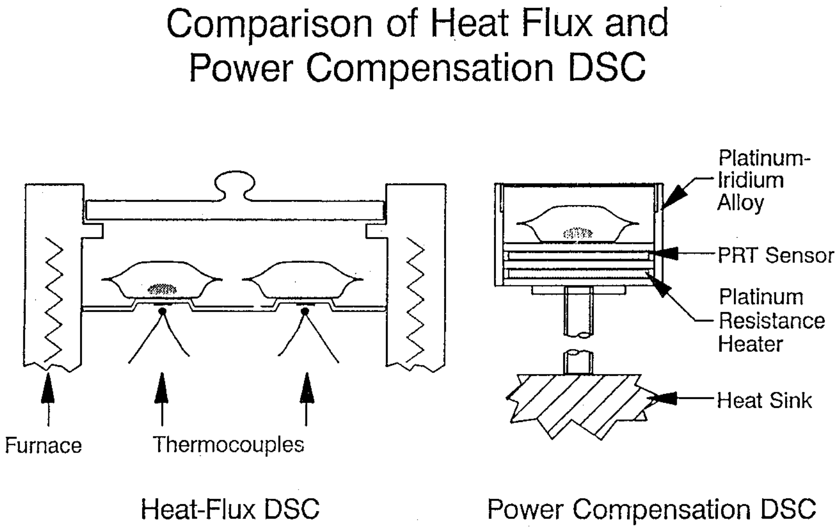


Fig. 6 Two most common types of DSC measuring cells. (Courtesy Perkin-Elmer Corporation)

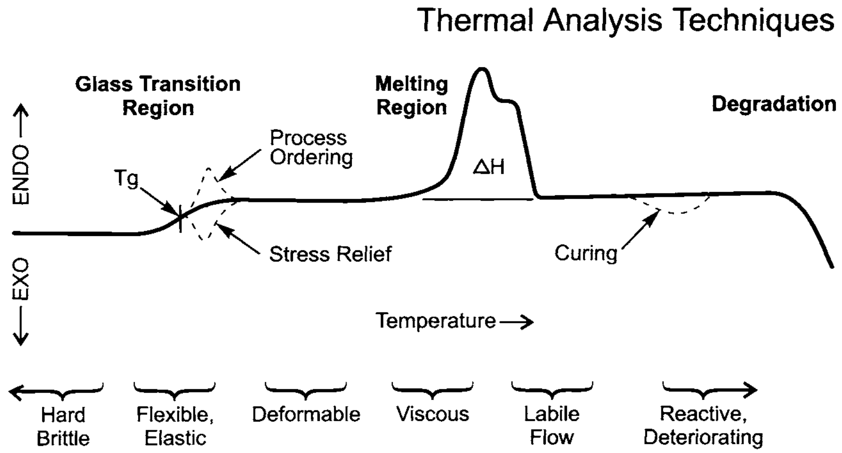


Fig. 7 Typical DSC thermogram. (Courtesy Perkin-Elmer Corporation)

ratio, and/or processing history. Often quality procedures involve comparing the melting profile to that of a standard "good" material.

When a sample cures, more energy is usually released, and the change is exothermic. The area of the curing peak is proportional to the number of cross-links that were formed. This indicates degree of cure. The shape of the curing curve can be analyzed to obtain the reaction kinetic parameters.

Thermogravimetric Analysis (TGA)

Thermogravimetric analysis is a test procedure in which changes in weight of a specimens is progressively heated. The sample weight is continuously monitored as the temperature is increased either at a constant rate or through a series of steps. The components of a polymer or elastomer formulation volatilize or decompose at different temperatures. This leads to a series of weight loss steps that allow the components to be quantitatively measured. A typical high-performance apparatus consists of an analytical balance supporting a platinum crucible for the specimen, the crucible situated in a electric furnace. Variations in instrumentation include horizontally mounted furnaces and top loading balances.

A simple thermal separation can be seen in the determination of mineral filler in polypropylene (Fig. 8). The sample is heated in an air atmosphere to completely decompose the polypropylene such that the remainder is mineral filler. The amount of volatiles (here, none), polymer, and filler is obtained by analysis of the weight loss curve. The small weight loss (3.5%) occurring over the temperature range of 750–800°C is characteristic of the particular mineral filler used in this sample. An unfilled polypropylene thermogram is also run to compare with the filled formulation. TGA is very useful in characterizing polymers containing different levels of additives by measuring the degree of weight loss. TGA can also be used to identify the ingredients of blended compounds according to the relative stability's of individual components. The thermal stability of a polymer can be obtained through a kinetic analysis of the decomposition profile.

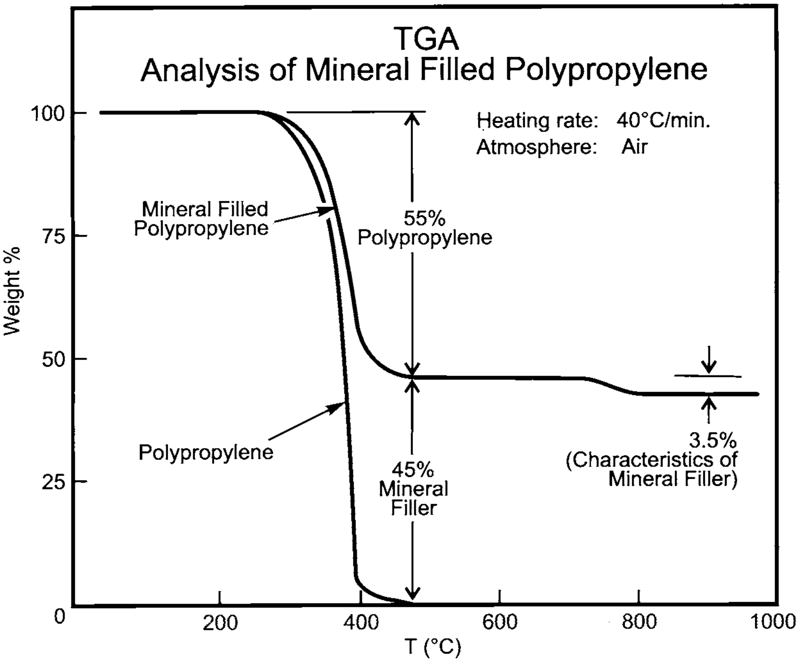


Fig. 8 Typical TGA thermogram. (Courtesy Perkin-Elmer Corporation)

Thermomechanical Analysis (TMA)

When a sample is heated, its dimensions change because of thermal expansion, stress reorientation, and deformation under an imposed stress. Thermomechanical analysis consists of measuring these properties using a constant force on the sample. Under a no-load or fixed load, it measures dimensional change in the vertical direction as the sample temperature is controlled. TMA equipment (Fig. 9) consists of a probe mechanically connecting three entities: (1) a force transducer to control the force applied to the sample, (2) a position transducer to measure the displacement, and (3) a temperature-controlled sample specimen. Samples to be examined are cut to a defined geometry and then deformed by the TMA probe in a defined manner. Sample deformation includes compression, tension, and bending geometries. Once mounted, the sample is surrounded by a furnace and monitored by a thermocouple.

A typical TMA thermal curve is shown in Fig. 10. Here a fused silica rod is used as a compression probe. The probe is placed onto the sample, and the thermal expansion or contraction is recorded as a function of temperature. The polymer used in the test contains a chemical blowing agent. Over the first 60°C, the TMA probe is slightly pushed up by the expansion of the polymer solid. As the melting point is reached, the lightly loaded probe penetrates the sample at a rate related to the viscosity. This penetration is later reversed by the expansion of the foam as the blowing agent is released. Polymer characterization through the use of TMA is accomplished by determining the glass transition temperature, the coefficient of expansion, and the elastic modulus. TMA data also correlates with the Vicat softening point and the heat distortion temperature.

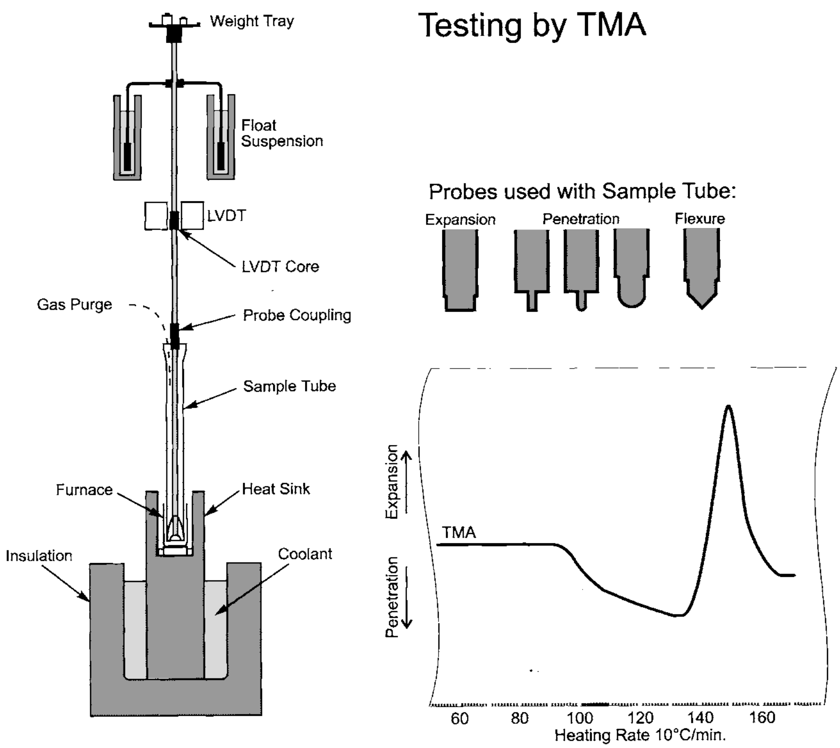


Fig. 9 Testing by TMA (schematic). (Courtesy Perkin-Elmer Corporation)

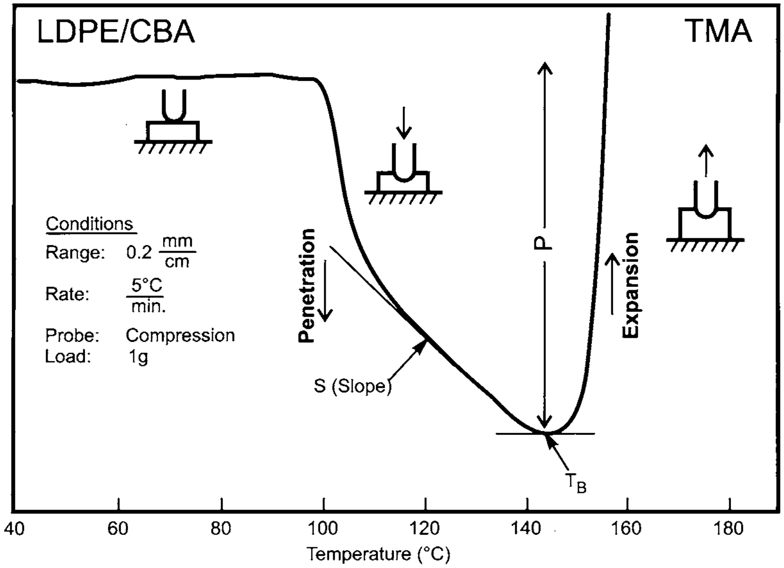


Fig. 10 Typical TMA thermal curve. (Courtesy Perkin-Elmer Corporation)

1.6 Spectroscopy

Infrared spectroscopy is one of the most widely used material analysis techniques for over 70 years. An infrared spectrum represents a fingerprint of a sample with absorption peaks that correspond to the frequencies of vibrations between the bonds of the atoms making up the material. Because each different material is a unique combination of atoms, no two compounds produce the exact same infrared spectrum. This fact allows a positive identification of polymeric materials. By studying the size of the peaks in the spectrum, one can also determine the amount of material present.

A newly developed technique called Fourier transform infrared (FT-IR) spectrometry overcomes the limitations encountered with the traditional infrared technique. The original infrared instruments were of dispersive type, which separated the individual frequencies of energy emitted from infrared source using a prism or grating. These instruments measured each frequency individually, making the entire process painfully slow. Modern FT-IR instruments can process as many as 100 samples per day compared to only 2–4 samples per day. FT-IR spectroscopy is fast, precise, and simple, requiring a very small amount of sample for a successful amalysis. In infrared spectroscopy, IR radiation is passed through a sample. Some of the infrared radiation is absorbed by the sample and some of it is passed through (transmitted). The resulting spectrum represents molecular absorption and transmission, creating a molecular fingerprint of the sample. Figure 11 graphically illustrates this process.

The sample analysis process is quite simple with the use of modern spectrometer coupled with a powerful computer. Infrared energy is emitted from a glowing black-body source. This beam passes through an interferometer where the ‘‘spectral encoding’’ takes place. The resulting interferogram signal then exits the interfermeter. Next, the beam enters the sample compartment where it is transmitted though or reflected off the surface of the sample. This is where the specific frequencies of energy, which are uniquely characteristic of the sample, are absorbed. The beam finally passes through a detector and the signal is sent to a computer where a mathematical technique called the Fourier transformation takes place. The infrared spectrum is displaced on the CRT for analysis and interpretation.

The computer is equipped with a collection of thousands of known polymer and additive FT-IR spectra for easy comparison and identification. A spectral library search can identify a polymer, additive, or a contaminant within minutes, making the entire process very fast and efficient. Figure 12 illustrates the entire FT-IR process.

2 IDENTIFICATION ANALYSIS OF PLASTIC MATERIALS

Plastic products are manufactured using a variety of processing techniques and materials. It is practically impossible to identify a plastic material or product by a visual inspection or a simple mechanical test. There are many reasons that necessitate the identification of plastics. One of the most common reasons is the need to identify plastic materials used in competitive products. Defective products returned from the field are quite often put through rigorous identification analysis. Sometimes it is necessary to identify a finished product at a later date

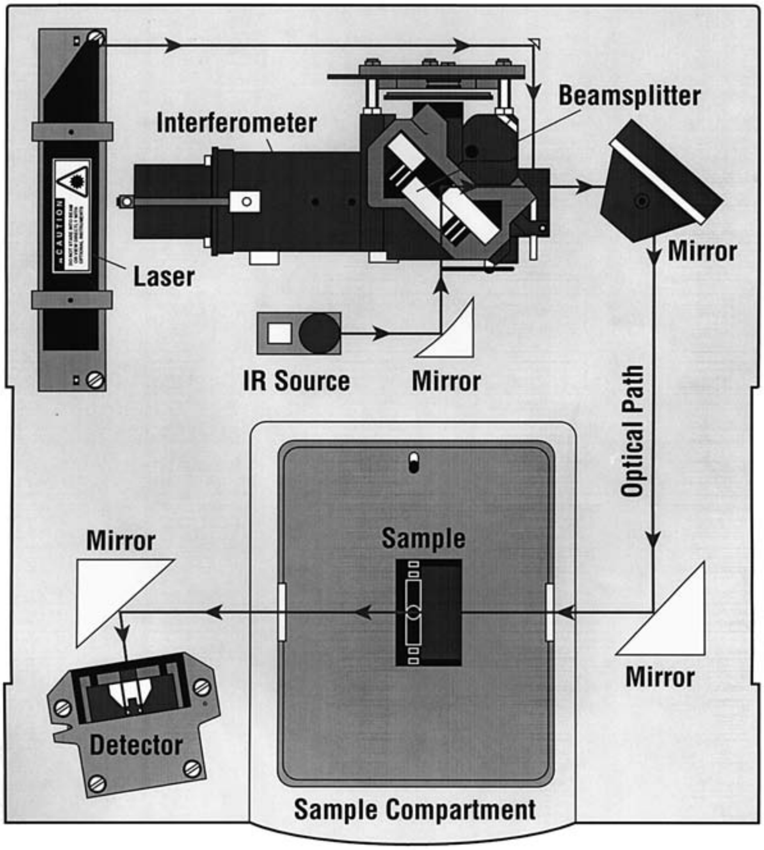


Fig. 11 Sample spectrometer layout. (Courtesy Nicolet Instruments)

in order to verify the material used during its manufacture. The custom compounders of reprocessed materials may also need to identify already processed material purchased from different sources. Quite often, processors find substantial quantities of plastic material, hot stamp foils, and decals in the warehouse without any labels to identify the particular type. A little knowledge of the identification process can save time and money.

On a rare occasion, the buyers of molded parts may choose to verify the material specified in the product by performing a simple identification analysis. The development of new material is another reason for such analysis.

There are two ways plastic materials can be identified. The first technique is simple, quick, and inexpensive. It requires very few tools and little knowledge of plastic materials. The second approach is to perform a systematic chemical or thermal analysis. The latter technique is very complex, time-consuming, and expensive. The results can only be interpreted by a person well versed in polymer chemistry. Plastic materials are often copolymerized, blended, and modified with filler or compounded with different additives such as flame retardants, blowing agents, lubricants, and stabilizers. In such cases, simple identification techniques will not yield satisfactory results. The only true means of positive identification is a complex chemical or thermal analysis.

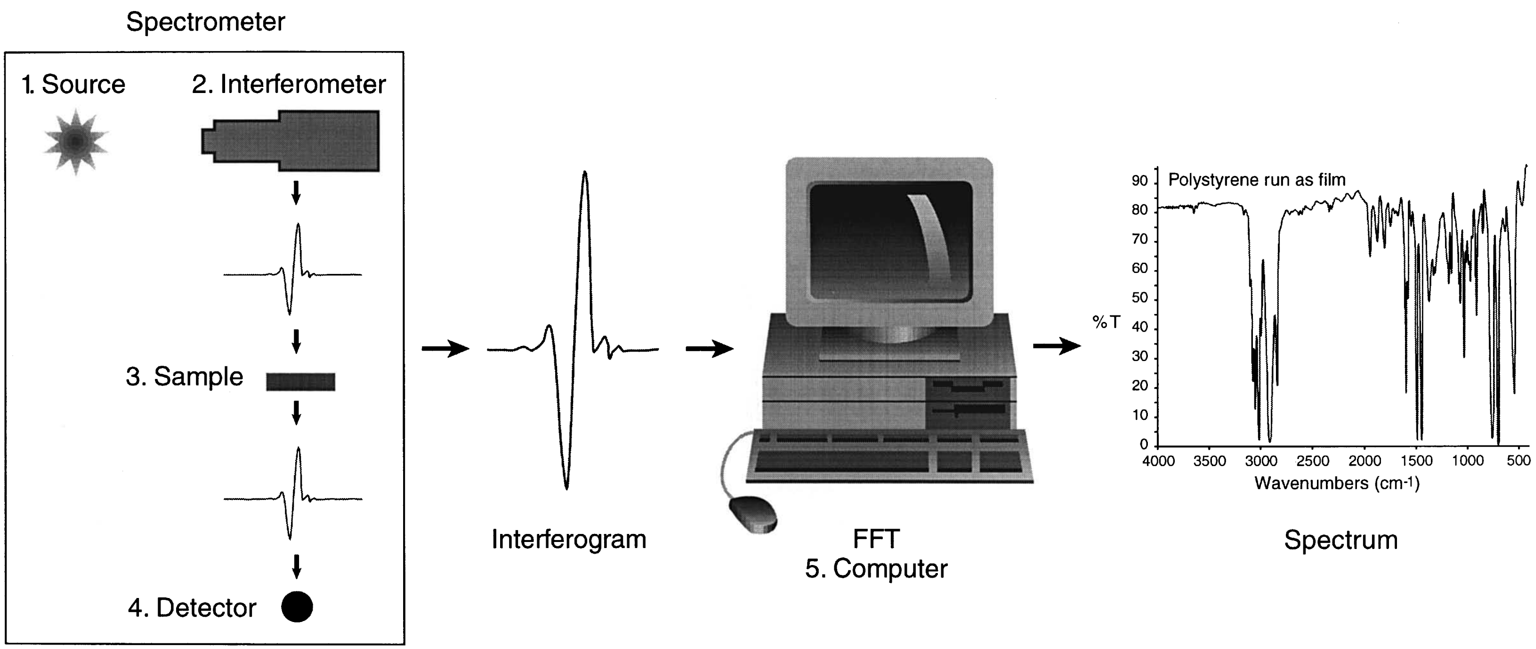


Fig. 12 Sample Analysis process. (Courtesy Nicolet Instruments)

The first technique is laid out in a flowchart (Fig. 13) for easy step-by-step identification by process of elimination. This is shown in the plastics identification chart. There are some basic guidelines one must follow to simplify the procedure. The first step is to determine whether the material is thermoplastic or thermoset. This distinction is made by simply probing the sample with a soldering iron or a hot rod heated to approximately 500°F. If the sample softens, the material is thermoplastic. If not, it is thermoset. The next step is to conduct a flame test. It is desirable to use a colorless Bunsen burner. A matchstick can also be used in place of a Bunsen burner. However, care must be taken to distinguish between the odor of the materials used in the match and the odor given off by burning plastic materials. Before commencing the burning test, it is advisable to be prepared to write down the following observations:

1. Does the material burn?
2. Color of flame.
3. Odor.
4. Does the material drip while burning?
5. Nature of smoke and color of smoke.
6. The presence of soot in the air.
7. Self-extinguishes or continues to burn.
8. Speed of burning—fast or slow.

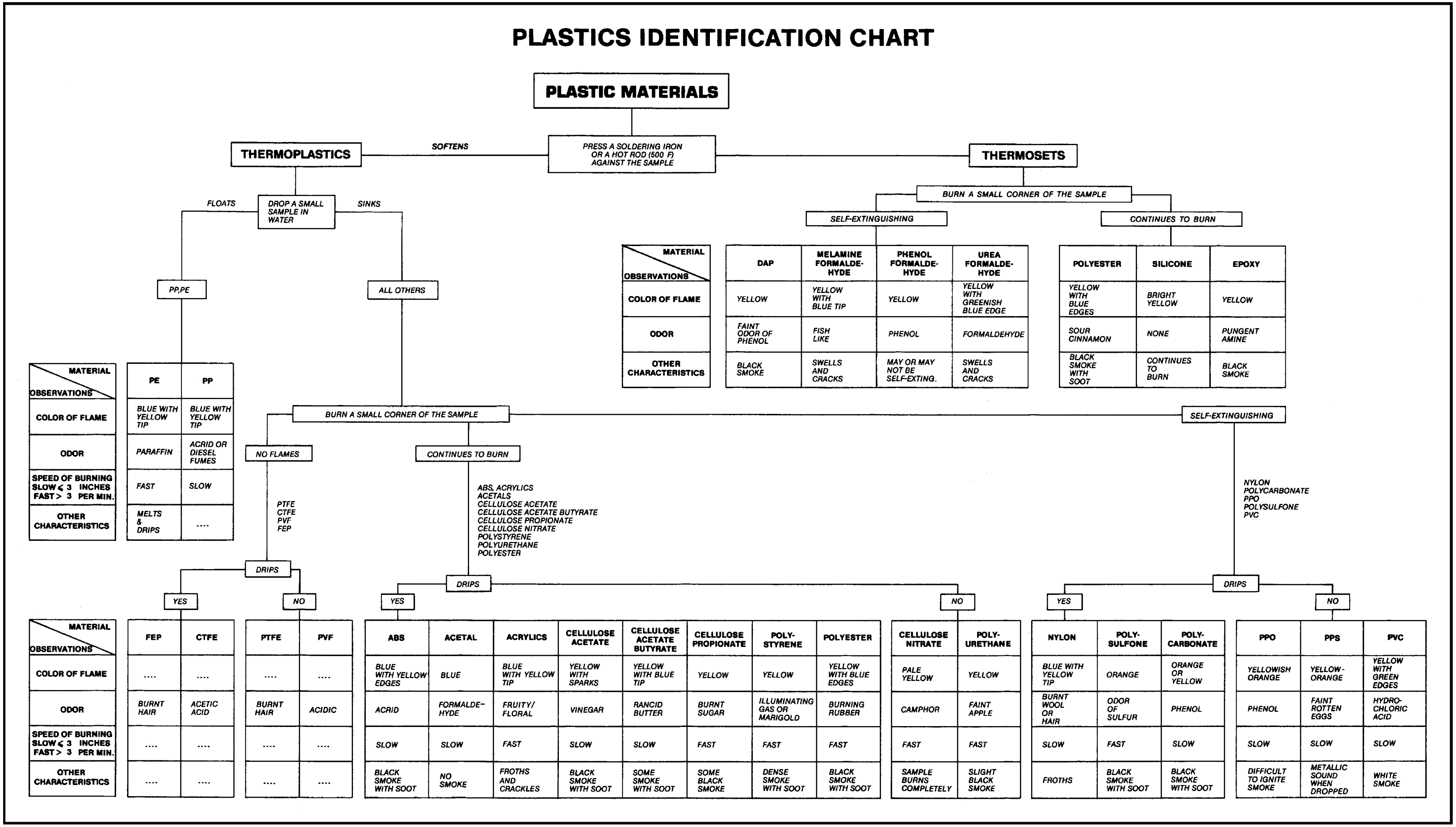
To identify the material, compare the actual observations with the ones listed in Fig. 13. The accuracy of the test can be greatly improved by performing similar tests on a known sample. While performing the identification tests, one must not overlook safety factors. The drippings from the burning plastic may be very hot and sticky. After extinguishing the flame, inhale the smoke very carefully. Certain plastics such as acetals give off a toxic formaldehyde gas that may cause a severe burning sensation in the nose and chest.

The results of the simple identification technique can be further confirmed by the following tests:

1. Melting point test
2. Solubility test
3. Copper wire test
4. Specific gravity test

2.1 Melting Point Determination

Two basic methods are used for melting point determination. For the first method, a Fisher–Johns melting point apparatus, as shown in Fig. 14, is most commonly used. The apparatus consists of a rheostatically controlled heated block, a thermometer, and a viewing magnifier. A small pellet or a sliver of the plastic material to be tested is placed on the electrically heated block along with a few drops of silicone oil. A cover glass is placed over the material and the heat is gradually increased until the sample material melts or softens enough to deform. The meniscus formed by the oil is viewed through the magnifier. The



MATERIAL / OBSERVATIONS	PE	PP
COLOR OF FLAME	BLUE WITH YELLOW TIP	BLUE WITH YELLOW TIP
ODOR	PARAFFIN	ACRID OR DIESEL FUMES
SPEED OF BURNING SLOW < 3 INCHES FAST > 3 PER MIN.	FAST	SLOW
OTHER CHARACTERISTICS	MELTS & DRIPS	....

MATERIAL / OBSERVATIONS	DAP	MELAMINE FORMALDE-HYDE	PHENOL FORMALDE-HYDE	UREA FORMALDE-HYDE	POLYESTER	SILICONE	EPOXY
COLOR OF FLAME	YELLOW	YELLOW WITH BLUE TIP	YELLOW	YELLOW WITH GREENISH BLUE EDGE	YELLOW WITH BLUE EDGES	BRIGHT YELLOW	YELLOW
ODOR	FAINT ODOR OF PHENOL	FISH LIKE	PHENOL	FORMALDEHYDE	SOUR CINNAMON	NONE	PUNGENT AMINE
OTHER CHARACTERISTICS	BLACK SMOKE	SWELLS AND CRACKS	MAY OR MAY NOT BE SELF-EXTING.	SWELLS AND CRACKS	BLACK SMOKE WITH SOOT	CONTINUES TO BURN	BLACK SMOKE

MATERIAL / OBSERVATIONS	FEP	CTFE	PTFE	PVF	ABS	ACETAL	ACRYLICS	CELLULOSE ACETATE	CELLULOSE ACETATE BUTYRATE	CELLULOSE PROPIONATE	POLY-STYRENE
COLOR OF FLAME	....	....	....	....	BLUE WITH YELLOW EDGES	BLUE	BLUE WITH YELLOW TIP	YELLOW WITH SPARKS	YELLOW WITH BLUE TIP	YELLOW	YELLOW
ODOR	BURNT HAIR	ACETIC ACID	BURNT HAIR	ACIDIC	ACRID	FORMALDE-HYDE	FRUITY/ FLORAL	VINEGAR	RANCID BUTTER	BURNT SUGAR	ILLUMINATING GAS OR MARIGOLD
SPEED OF BURNING SLOW < 3 INCHES FAST > 3 PER MIN.	....	....	....	....	SLOW	SLOW	FAST	SLOW	SLOW	FAST	FAST
OTHER CHARACTERISTICS	....	....	....	....	BLACK SMOKE WITH SOOT	NO SMOKE	FROTHS AND CRACKLES	BLACK SMOKE WITH SOOT	SOME SMOKE WITH SOOT	SOME BLACK SMOKE	DENSE SMOKE WITH SOOT

MATERIAL / OBSERVATIONS	POLYESTER	CELLULOSE NITRATE	POLY-URETHANE	NYLON	POLY-SULFONE	POLY-CARBONATE	PPO	PPS	PVC
COLOR OF FLAME	YELLOW WITH BLUE EDGES	PALE YELLOW	YELLOW	BLUE WITH YELLOW TIP	ORANGE	ORANGE OR YELLOW	YELLOWISH ORANGE	YELLOW-ORANGE	YELLOW WITH GREEN EDGES
ODOR	BURNING RUBBER	CAMPHOR	FAINT APPLE	BURNT WOOL OR HAIR	ODOR OF SULFUR	PHENOL	PHENOL	FAINT ROTTEN EGGS	HYDRO-CHLORIC ACID
SPEED OF BURNING SLOW < 3 INCHES FAST > 3 PER MIN.	FAST	FAST	FAST	SLOW	FAST	SLOW	SLOW	SLOW	SLOW
OTHER CHARACTERISTICS	BLACK SMOKE WITH SOOT	SAMPLE BURNS COMPLETELY	SLIGHT BLACK SMOKE	FROTHS	BLACK SMOKE WITH SOOT	BLACK SMOKE WITH SOOT	DIFFICULT TO IGNITE SMOKE	METALLIC SOUND WHEN DROPPED	WHITE SMOKE

Fig. 13 Plastics identification chart (© Vishu Shah, www.consultekusa.com).

Fig. 14 Melting point determination.

temperature at which the meniscus moves is considered the melting point. The expected accuracy of the test is within ± 5°F of the published literature value. This method can be used for both crystalline and amorphous plastics. All crystalline plastics have a sharp melting point and the transition is much easier to detect. In contrast, amorphous plastics melt over a wide range and an exact melting point is difficult to determine.

The second method, known as the Kofler method, is used only for semicrystalline polymers. It consists of heating the sample by a hot-stage unit mounted under a microscope and viewing it between crossed polarizers. When crystalline material melts, the characteristic double refraction from the crystalline aggregates disappears. The point at which the double refraction or birefringence (typically a rainbow color) completely disappears is taken as the melting point of the polymer. The use of a control sample for comparison is particularly helpful in both methods.

2.2 Solubility Test

The behavior of plastic materials in various organic solvents often indicates the type of material. The solubility data found in the literature is of a general nature and consequently difficult to use at times. A partial solubility of some plastics in different solvents and a high concentration of additives such as plasticizers further complicate identification by the solubility test. However, a solubility test is very useful in distinguishing between the different types of the same base polymer. For example, cellulose acetate can be distinguished from cellulose acetate butyrate because the acetate is completely soluble in furfuryl alcohol whereas the butyrate is only partially soluble. Types of nylons and polystyrenes can be identified similarly. The solubility test is best conducted by placing a sliver of the sample in a small test tube, adding the solvent, and gently stirring it. Ample time should be allowed before passing judgment regarding solubility of the sample in a particular solvent.

2.3 Copper Wire Test

The presence of chlorine such as in polyvinyl chloride can be easily confirmed by simply conducting the copper wire test. The tip of the copper wire should be heated to a red-hot temperature in a flame. A small quantity of material is picked up by drawing the wire across the surface of the sample. The tip of the wire is returned to the flame. A green-colored flame indicates the presence of chlorine in the material. Fluorocarbons can also be identified by detecting the presence of fluorine.

2.4 Specific Gravity Test

The increasing use of plasticizers, fillers, reinforcing agents, and other additives makes the identification of plastics by the specific gravity test very difficult.

2.5 Chemical and Thermal Analysis for Identification of Polymers

The following techniques are those most commonly used today to positively identify plastic materials and additives. Table 2 lists the identification technique along with the proper application.

1. Fourier transform infrared analysis (FT-IR)
2. Gas chromatography (GC)
3. Thermogravimetric analysis (TGA)
4. Differential scanning calorimetry (DSC)
5. Thermomechanical analysis (TMA)
6. Nuclear magnetic resonance (NMR)
7. X-ray analysis
8. Pyrolysis
9. Liquid and gel permeation chromatography

Table 2 Identification Techniques for Polymer and Additives

Technique	Identification
LC/GPC	Polymer molecular weight distribution. Phenols, phosphites, plasticizers, lubricants
GC	Residual monomers Nonpolymeric compounds Plasticizers
IR	Polymer type Additives
Thermal	Fillers Lubricants Polymer molecular weight
X-ray	Fillers Flame retardants Stabilizers
NMR	Polyesters Silicones Phenols
Wet chemistry	Lubricants Flame retardants Catalysts

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CHAPTER 22

PROFESSIONAL AND TESTING ORGANIZATIONS

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1 AMERICAN NATIONAL STANDARDS INSTITUTE (ANSI) 615
2 AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM) 616
3 FOOD AND DRUG ADMINISTRATION (FDA) 616
4 NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY (NIST) 617
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1 AMERICAN NATIONAL STANDARDS INSTITUTE (ANSI)

In 1918, when ANSI was founded, standardization activities were just beginning in the United States. Many groups were developing standards and their interests and activities overlapped. The standards they produced often duplicated or conflicted with each other. The result was the waste of manpower, money, and considerable confusion. Five professional/technical societies and three government departments decided a coordinator was needed and created ANSI to handle the job.

ANSI is a federation of standards competents from commerce and industry, professional, trade, consumer, and labor organizations and government. ANSI, in cooperation with these federation participants:

1. Identifies the needs for standards and sets priorities for their completion.
2. Assigns development work to competent and willing organizations.

Handbook of Materials Selection. Edited by Myer Kutz

3. Sees to it that public interests, including those of the consumer, are protected and represented.
4. Supplies standards writing organizations with effective procedures and management services to ensure efficient use of their manpower and financial resources and timely development of standards.
5. Follows up to assure that needed standards are developed on time.

Another role is to approve standards as American National Standards when they meet consensus requirements. It approves a standard only when it has verified evidence presented by a standards developer that those affected by the standard have reached substantial agreement on its provisions. ANSI's other major roles are to represent U.S. interests in nongovernmental international standards work, to make national and international standards available, and to inform the public of the existence of these standards.

2 AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

The American Society for Testing and Materials was founded in 1898. It is a scientific and technical organization formed for ''the development of standards on characteristics and performance of materials, products, systems and services and the promotion of related knowledge.'' ASTM is the world's largest source of voluntary consensus standards. The society operates through more than 135 main technical committees with 1550 subcommittees. These committees function in prescribed fields under regulations that ensure balanced representation among producers, users, and general-interest participants. The society currently has 32,000 active members, of whom approximately 17,000 serve as technical experts on committees, representing 76,200 units of participation.

Membership in the society is open to all concerned with the fields in which ASTM is active. An ASTM standard represents a common viewpoint of those parties concerned with its provisions, namely, producers, users, and general-interest groups. It is intended to aid industry, government agencies, and the general public. The use of an ASTM standard is purely voluntary. It is recognized that, for certain work or in certain regions, ASTM specifications may be either more or less restrictive than needed. The existence of an ASTM standard does not preclude anyone from manufacturing, marketing, or purchasing products or using products, processes, or procedures not conforming to the standard. Because ASTM standards are subject to periodic reviews and revision, it is recommended that all serious users obtain the latest revision.

A new edition of the *Book of Standards* is issued annually. On the average, about 30% of each part is new or revised. In 1996, the annual book of ASTM standards, which consisted of 71 parts and over 70,000 pages, included over 10,000 ASTM standards and tentatives.

3 FOOD AND DRUG ADMINISTRATION (FDA)

The Food and Drug Administration, first established in 1931, is an U.S. government agency of the Department of Health and Human Services. The FDA's activities are directed toward protecting the health of the nation against impure and unsafe foods, drugs, and cosmetics and other potential hazards.

The plastics industry is mainly concerned with the Bureau of Foods, which conducts research and develops standards on the composition, quality, nutrition, and safety of foods, food additives, colors, and cosmetics and conducts research designed to improve the detection, prevention, and control of contamination. The FDA is concerned about indirect additives. Indirect additives are those substances capable of migrating into food from contacting plastic materials. Extensive tests are carried out by the FDA before issuing safety clearance to any plastic material that is to be used in food contact applications. Plastics used in medical devices are tested with extreme caution by the FDA's Bureau of Medical Devices, which develops FDA policy regarding safety and effectiveness of medical devices.

Field operations for the enforcement of the laws under the jurisdiction of the FDA are carried out by 11 regional field offices, 22 district offices, and 124 resident inspection posts.

4 NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY (NIST)

The National Bureau of Standards (NBS) was established by act of Congress in March 1901. NBS was renamed the National Institute of Standard and Technology in 1988. The bureau's overall goal is to strengthen and advance the nation's science and technology and to facilitate their effective application for public benefit.

The bureau conducts research and provides a basis for the nation's physical measurement system, scientific and technological services for industry and government, a technical basis for increasing productivity and innovation, promoting international competitiveness in American industry, maintaining equity in trade and technical services, and promoting public safety. The bureau's technical work is performed by the National Measurement Laboratory, the National Engineering Laboratory, and the Institute for Computer Sciences and Technology.

5 NATIONAL ELECTRICAL MANUFACTURERS ASSOCIATION (NEMA)

The National Electrical Manufacturers Association was founded in 1926. This 600-member association consists of manufacturers of equipment and apparatus for the generation, transmission, distribution, and utilization of electric power. The membership is limited to corporations, firms, and individuals actively engaged in the manufacture of products included within the product scope of NEMA product subdivisions.

NEMA develops product standards covering such matters as nomenclature, ratings, performance, testing, and dimensions. NEMA is also actively involved in developing National Electrical Safety Codes and advocating their acceptance by state and local authorities. Along with a monthly news bulletin, NEMA also publishes manuals, guidebooks, and other material on wiring, installation of equipment, lighting, and standards. The majority of NEMA standardization activity is in cooperation with other national organizations. The manufacturers of wires and cables, insulating materials, conduits, ducts, and fittings are required to adhere to NEMA standards by state and local authorities.

6 NATIONAL FIRE PROTECTION ASSOCIATION (NFPA)

The National Fire Protection Association was founded in 1896 with the objective of developing, publishing, and disseminating standards intended to minimize the possibility and effect of fire and explosion. NFPA's membership consists of individuals from business and industry, fire service, health care, insurance, educational, and government institutions. NFPA conducts fire safety education programs for the general public and provides information on fire protection and prevention. Also provided by the association is the field service by specialists on flammable liquids, electricity, gases, and marine problems.

Each year, statistics on causes and occupancies of fires and deaths resulting from fire are compiled and published. NFPA sponsors seminars on the Life Safety Codes, National Electrical Code, industrial fire protection, hazardous materials, transportation emergencies, and other related topics. NFPA also conducts research programs on delivery systems for public fire protection, arson, residential fire sprinkler systems, and other subjects. NFPA publications include *National Fire Codes Annual, Fire Protection Handbook, Fire Journal*, and *Fire Technology*.

7 NATIONAL SANITATION FOUNDATION (NSF)

The National Sanitation Foundation, more commonly known as NSF, is an independent, nonprofit environmental organization of scientists, engineers, technicians, educators, and analysts. NSF frequently serves as a trusted neutral agency for government, industry, and consumers, helping them to resolve differences and unite in achieving solutions to problems of the environment.

At NSF, a great deal of work is done on the development and implementation of NSF standards and criteria for health-related equipment. Standard No. 1, concerning soda fountain and luncheonette equipment, was adopted in 1952. Since then, many new standards have been developed and successfully implemented. The majority of NSF standards relate to water treatment and purification equipment, products for swimming pool applications, plastic pipe for potable water as well as drain, waste, and vent (DWV) uses, plumbing components for mobile homes and recreational vehicles, laboratory furniture, hospital cabinets, polyethylene refuse bags and containers, aerobic waste treatment plants, and other products related to environmental quality.

Manufacturers of equipment, materials, and products that conform to NSF standards are included in official listings, and these producers are authorized to place the NSF seal on their products. Representatives from NSF regularly visit the plants of manufacturers to make certain that products bearing the NSF seal do indeed fulfill applicable NSF standards.

8 SOCIETY OF PLASTICS ENGINEERS (SPE)

The Society of Plastics Engineers was founded in 1942 with the objective of promoting scientific and engineering knowledge relating to plastics. SPE is a professional society of plastics scientists, engineers, educators, students, and others interested in the design, development, production, and utilization of plastics materials, products, and equipment. SPE currently has over 37,000 members scattered among its 91 sections. The individual sections as well as the SPE main body arranges and conducts monthly meetings, conferences, educational seminars, and plant tours throughout the year. SPE also publishes *Plastics Engineering, Polymer Engineering and Science, Plastics Composites*, and the *Journal of Vinyl Technology*. The society presents a number of awards each year encompassing all levels of the organization, section, division, committee, and international. SPE divisions of interest are color and appearance, injection molding, extrusion, electrical and electronics, thermoforming, engineering properties and structure, vinyl plastics, blow molding, medical plastics, plastics in building, decorating, mold making, and mold design.

9 SOCIETY OF PLASTICS INDUSTRY (SPI)

The Society of Plastics Industry is a major society, whose membership consists of manufacturers and processors of plastics materials and equipment. The society has four major operating units consisting of the Eastern Section, the Midwest Section, the New England Section, and the Western Section. SPI's Public Affairs Committee concentrates on coordinating and managing the response of the plastics industry to issues such as toxicology, combustibility, solid waste, and energy. The Plastic Pipe Institute is one of the most active divisions, promoting the proper use of plastic pipes by establishing standards, test procedures, and specifications. Epoxy Resin Formulators Division has published over 30 test procedures and technical specifications. Risk management, safety standards, productivity, and quality are a few of the major programs undertaken by the machinery division. SPI's other divisions include Expanded Polystyrene Division, Fluoropolymers Division, Furniture Division, International Division, Plastic Bottle Institute, Machinery Division, Molders Division, Mold Makers Division, Plastic Beverage Container Division, Plastic Packaging Strategy Group, Polymeric Materials Producers Division, Polyurethane Division, Reinforced Plastic/ Composites Institute, Structural Foam Division, Vinyl Siding Institute, and Vinyl Formulators Division.

The National Plastics Exposition and Conference, held every 3 years by the Society of Plastic Industry, is one of the largest plastic shows in the world. SPI works very closely with other organizations such as ASTM and ANSI to develop new test methods, standards, and specifications.

10 UNDERWRITERS LABORATORIES (UL)

Underwriters Laboratories, founded in 1894, is chartered as a not-for-profit organization to establish, maintain, and operate laboratories for the investigation of materials, devices, products, equipment, constructions, methods, and systems with respect to hazards affecting life and property.

There are five testing facilities in the United States and over 200 inspection centers. More than 700 engineers and 500 inspectors conduct tests and follow-up investigations to ensure that potential hazards are evaluated and proper safeguards provided. UL has six basic services it offers to manufacturers, inspection authorities, or government officials. These are product listing service, classification service, component recognition service, certificate service, inspection service, and fact finding and research.

UL's Engineering Services Department is in charge of evaluating individual plastics and other products using plastics as components. Engineering Services evaluates consumer products such as TV sets, power tools, appliances, and industrial and commercial electrical equipment and components. For a plastic material to be recognized by UL, it must pass a variety of UL tests including the UL 94 flammability test and the UL 746 series, short- and long-term property evaluation tests. When a plastic material is granted recognized component status, a yellow card is issued. The card contains precise identification of the material including supplier, product designation, color, and its UL 94 flammability classification at one or more thicknesses. Also included are many of the property values such as relative temperature index, hot wire ignition, high-current arc ignition, and arc resistance. These data also appear in the *Plastics Recognized Component Directory*.

UL publishes the names of the companies who have demonstrated the ability to provide a product conforming to the established requirements, upon successful completion of the investigation and after agreement of the terms and conditions of the listing and follow-up service. Listing signifies that production samples of the product have been found to comply with the requirements, and that the manufacturer is authorized to use the UL Listing Mark on the listed products that comply with the requirements.

UL's consumer advisory council was formed to advise UL in establishing levels of safety for consumer products, to provide UL with additional user field experience and failure information in the field of product safety, and to aid in educating the general public in the limitations and safe use of specific consumer products.

11 TYPICAL COSTS FOR TESTING SERVICES

Due to the complexity of the tests, varying requirements and other circumstances, it is very difficult to estimate the cost for a particular analytical test. However, an attempt is made to give reader a general idea of the typical costs for testing services in Table 1.

Table 1 Typical Costs for Testing Services

Type of Tests[a]	Cost per Test
Analytical: FT-IR, DSC, TGA, TMA, GPC, Additives identification, filler content, carbon content, moisture content, chromatography, spectrometry, NMR	\$100–\$500
Mechanical/Physical: Tensile, flexural, compressive, shear, puncture, tear, Poisson's ratio, creep, impact testing, hardness, abrasion, specific gravity, chemical compatibility, density	\$100–\$400
Electrical: Dielectric constant, dissipation factor, volume resistivity, surface resistivity, dielectric strength, EMI/RFI shielding effectiveness, arc resistance	\$100–\$400
Thermal/Flammability: Coefficient of thermal expansion, thermal conductivity, TMA, DSC, heat deflection temperature, melting point determination, flammability tests, oxygen index, TGA	\$100–\$500
Rheological: Capillary rheometer tests, viscosity tests, melt flow index, intrinsic viscosity, brookfield viscosity, die swell measurements	\$100–\$300
Optical: Colorimeter, spectrophotometer, color measurement, UV tests, light absorption and transmission, refractive index, yellowness index, gloss	\$100–\$300

Source: Plastics Technology Laboratories, Inc. Pittsfield, MA; www.ptli.com.

[a] FTIR, Fourier Transform Infrared Spectrometry
DSC, Differential Scanning Calorimetry
TGA, Thermogravimetric Analysis
TMA, Thermomechanical Analysis
GPC, Gel Permeation Chromatography
NMR, Nuclear Magnetic Resonance

12 INDEPENDENT TESTING LABORATORIES

Atlas Weathering Services Group
17301 Okeechobee Road
Miami, Florida 33015
(800) 255-3738

Battelle Memorial Institute
505 King Avenue
Columbus, Ohio 43201
(614) 424-6533

Broutman, L.J., Associates Ltd.
3424 South State St.
Chicago, Illinois 60616
(312) 842-4100

Chemir/Polytech Laboratories, Inc.
2672 Metro Boulevard
St. Louis, Missouri 63043
(314) 291-6620

CRT Labs Inc., (Calif. Resin Testing)
2812 Walnut, Suite A
Tustin, California 92680
(800) 597-5227

Delsen Testing Laboratories, Inc.
1024 Grand Central Avenue
Glendale, California 91201
(213) 245-8517

Detroit Testing Laboratory, Inc.
7111 E. Eleven Mile Road
Warren, Michigan 48092-2709
(810) 754-9000

Dynepco Inc.
84 N. Main Street
Wharton, New Jersey 07885
(201) 361-8900

Engineered Polymers
P.O. Box 360, 42 Sears Way, West
Springfield, Massachusetts 01090
(413) 787-0780

Gaynes Testing Laboratories, Ltd.
1642 W. Fulton Street
Chicago, Illinois 60612
(312) 421-5257

Ghesquire Plastic Testing, Inc.
20450 Harper Avenue
Harper Woods, Michigan 48225
(313) 885-3535

Hunter Associates Laboratory, Inc.
11491 Sunset Hills Road
Reston, Virginia 22090
(703) 471-6870

Integrated Design, Inc.
10 River Road, Suite 101
Uxbridge, Massachusetts 01569-2245
(800) 399-8457

Measurement Technology Corp.
(Measure Tech)
4396 Round Lake Road West
St. Paul, Minnesota 55112-3923
(612) 633-7651

Measurement Technology Inc.
4240 Lock Highland Parkway
Roswell, Georgia 30075
(770) 587-2222

Monarch Analytical Laboratories, Inc.
349 Tomahawk Drive
Maumee, Ohio 43537
(419) 897-9000

Plastics Technology Laboratories, Inc.
50 Pearl Street
Pittsfield, Massachusetts 01201
(413) 499-0983

Polyhedron Laboratories, Inc.
10626 Kinghurst Street
Houston, Texas 77099
(713) 879-8600

Polymer Solutions Inc.
1872 Pratt Drive
Blacksburg, Virginia 24060-6363
(540) 231-3058

Q-Panel Lab Products
26200 First Street
Cleveland, Ohio 44145
(216) 835-8700

Rutherford Research Corp.
P.O. Box 249
Rutherford, New Jersey 07070
(201) 933-2091

Scientific Process and Research, Inc.
67 Veronica Avenue
Somerset, New Jersey 08873
(908) 846-3477

SGS U.S. Testing Co., Inc.
291 Fairfield Avenue
Fairfield, New Jersey 07004
(800) 777-8378

Skeist Inc.
375 Route 10
Whippany, New Jersey 07891
(201) 512-2020

Specialty Testing and Equipment, Inc.
42 Dutch Mill Road
Ithaca, New York 14850
(607) 257-1532

Springborn Laboratories, Inc.
10 Springborne Center
Enfield, Connecticut 06082
(860) 749-8371

Underwriters Laboratories
333 Pfingsten Road
Northbrook, Illinois 60062
(847) 272-8800

University of Massachusetts Lowell, Inst. Plastics Innov.
333 Aiken Street
Lowell, Massachusetts 01854-2881
(508) 934-3445

VTEC Laboratories, Inc.
212 Manida Street
Bronx, New York 10474
(718) 542-8248

X-Rite, Inc.
3100 44th Street
S.W. Grandville, Michigan 49418
(800) 545-0694

CHAPTER 23
CERAMICS TESTING

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1 INTRODUCTION

Ceramics are being used in an ever-increasing capacity in numerous industrial and scientific applications. Advanced ceramics, in particular, have great future possibilities for use in a wide variety of applications where their unique combination of properties can achieve better results than other materials such as metals and polymers. Advanced ceramics are typically wear and corrosion resistant, lightweight, and thermodynamically stable. In addition, many advanced ceramics have electrical properties that make them advantageous for use in electronic applications such as electronic packaging. Ceramics are finding increasing use in such areas as biomaterials, electronics, MEMS (microelectromechanical systems), and heat transfer applications such as gas turbines in the aerospace industry. The market value of advanced ceramics in the United States is estimated to increase to \$11 billion by the year 2003 from a value of \$7.5 billion in 1998.[1] Similarly, the world demand for advanced ceramics has increased from an estimated \$16.7 billion in 1994 to \$25.3 billion in the year 2000.[2]

Handbook of Materials Selection. Edited by Myer Kutz

With the steadily increasing use of ceramics in industry, there exists an increasing demand to characterize and quantify the properties of ceramics. This leads to higher demand for improved testing techniques to yield more exact data used for endeavors such as design, safety analysis, quality control, and scientific understanding. In particular, the brittle nature of ceramics makes their fracture characteristics especially important for the engineer that needs to design ceramic components with life-cycle and safety considerations. In addition to the predominant mechanical testing being performed, there is demand for improved testing of thermal, electrical, and environmental properties as well as others.

Of equal importance to the driving forces behind ceramic testing is the value of the data being obtained from these tests. Do the potential benefits of the data outweigh the cost of performing the tests? What are the predictive abilities of the quantified test results?

To aid the engineer or scientist who is starting his or her research into potential ceramic test methods, this chapter gives a brief overview of the major test methods currently being employed, relative advantages and disadvantages to each other, and some common sources of error. In addition, a listing of standards that are both relevant and directly applicable to ceramics is given for various testing methods. These standards can be further investigated by the reader who wants a more in-depth look into the exact setup and requirements of a given test. Standardization is an important process in the evolution of ceramic testing to improve accuracy and precision of measured results. New standards are continuously being developed and while not all the standards listed for each test are specifically targeted for ceramics, they still have relevance in terms of improving the exactness of the test setup and results.

2 MECHANICAL TESTING

With the increasing role of ceramics in technology, further understanding of mechanical properties has become increasingly more important. This has resulted in the use and standardization of various test methods to better understand and quantify mechanical properties. Test methods reveal such properties as strength, fatigue, fracture resistance, creep, and slow crack growth, which contribute to design, scientific understanding, and estimations of service life among others.

2.1 Strength

One of the most important properties for characterizing a material is strength. Strength (yield, ultimate, etc.) is often used as a measure of success in materials development.[3] The characterization of the fracture strength (approximately equal to ultimate strength for brittle materials) distribution is needed when ceramic design for structural applications involves failure probability as a criteria.[4] Various test methods are employed to determine the fracture strength of a given ceramic material. Usually, the fracture strength is equated to the maximum stress at the point of fracture, which requires that the stress distribution in the test specimen be known. A common source of error in tests measuring fracture strength is that the strength of ceramic materials is strongly influenced by the test specimen's size, geometry, and surface finish. The more common methods for measuring fracture strength include uniform uniaxial stress loading, nonuniform uniaxial stress loading, and biaxial stress loading.

Uniform uniaxial stress load testing is performed by putting a given test specimen into a state of tension or compression (Fig. 1). Testing in a uniform, uniaxial stress field controls the stress-state variable that allows determination of the mechanical behavior at given loads. Tension and compression tests are not as common as the nonuniform flexure test because the brittle nature of many ceramics results in the need for specialized testing equipment, extensive preparation of test specimen, and an adequately uniform stress state, which can be difficult to achieve.[3]

Tension

The tensile test equipment consists of two main parts, the test specimen grip holding the actual test specimen and the interface attachment that connects the test specimen grip to the test machine. The grip should be designed to reduce any eccentricity in order to maintain a uniaxial stress state. Ceramics, in particular, are not able to use a threaded head as an interface because of the difficult nature of machining such a brittle material. The tapered head test specimen is also difficult to machine to proper tolerances. The button head test specimen has emerged as the most reliable interface method.[3] The attachment interfacing between the test specimen grip and the test machine is usually one of two designs: flexible, self-aligning and fixed, alignable.[3]

Ideally, the stress state will be characterized by the simple equation:

$$\sigma = \frac{P}{A}$$

where σ is the normal stress, P is the applied force, and A is the cross sectional area. However, the actual stress state in the gauge section, σ_{gs}, will include error from eccentricity in the testing equipment and/or the test specimen and can be characterized as:

$$\sigma_{\mathrm{gs}} = \sigma_a + \sigma_b$$

where

$$\text{axial stress, } \sigma_a = \frac{P}{A} \quad \text{and} \quad \text{bending stress, } \sigma_b = \frac{Per}{I}$$

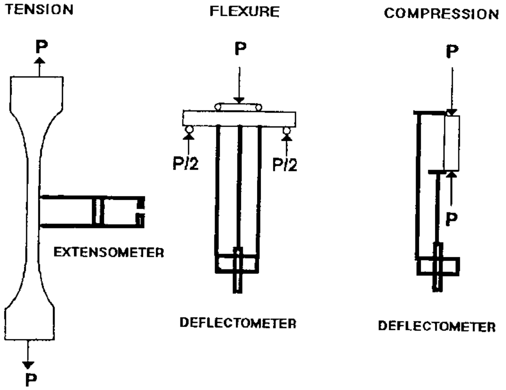

Fig. 1 Schematic of tensile, flexure, and compressive setups. (From Ref. 3 by courtesy of Marcel Dekker Inc.)

with *e* being the eccentricity distance, *r* being the distance from the point in the gauge section at which the stress is being measured to the centroid, and *I* being the moment of inertia in the gauge section cross section.

Some of the tensile testing standards that apply directly to ceramics are:

Japanese Industrial Standards Committee (JISC),* "Testing Methods for Tensile Strength of Monolithic Advanced Ceramics at Room and Elevated Temperature," JIS R1606-1995

American Society for Testing and Materials (ASTM),† "Standard Test Method for Tensile Strenth of Monolithic Advanced Ceramics at Ambient Temperatures," C1273-95

ASTM, "Standard Test Method for Tensile Strength of Monolithic Advanced Ceramics at Elevated Temperatures," C1366-97

The following standards are directly applicable to metals but can have applicability to ceramics:

ASTM, "Standard Test Methods for Tension Testing of Metallic Materials," E8-96a

ASTM, "Standard Test Methods for Elevated Temperature Tension Testing of Metallic Materials," E21-92

ASTM," Standard Practice for Verification of Specimen Alignment under Tensile Loading," E1012-93A

Compression

The compression strength of ceramic materials is usually much greater than that in tension. Consequently, tensile strength is usually the critical factor in terms of design. The compression test usually consists of two load blocks exerting a compressive force on a cylindrical test specimen. The test specimen should be uniform to avoid buckling of individual layers aligned with the applied load.

Common sources of error are size mismatches between the load block and specimen, surface irregularities, and eccentric loading as explained in the tensile testing section.[3] The first three errors bring about excessive stresses in the ends of the test specimen, which can cause failures in the nongauge section of the test specimen. Eccentricity, as in tensile testing, reduces uniformity in the gauge section stress state.

The following standards are directly applicable to compressive testing of ceramics:

JISC, "Testing Method for Compressive Strength of High Performance Ceramics," R1608-1990

ASTM, "Standard Test Method for Compressive (Crushing) Strength of Fired Whiteware Materials," C773-88

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* Japan Standards Association, Tokyo, Japan.

† ASTM International, West Conshohocken, Pennsylvania, USA.

Flexure

Testing in flexure typically involves a three- or four-point loading of a test specimen as shown in Fig. 2. Compared to tension and compression tests, flexure tests are less expensive, simpler in setup, and easier to adapt to elevated temperature testing.[3] The applied moment, M, yields the equation for uniaxial normal stress, σ:

$$\sigma = \frac{MC}{I}$$

where the moment of inertia I is $(bh^3)/12$ (b = width and h = height) and C is the distance from the neutral axis to the outer surface of the test specimen. Given that the moment $M = P(L_o - L_i)/4$ (L_o = outer span, L_i = inner span) and $y = h/2$ we can substitute into the equation to find the fracture strength, S_f, which is the maximum tensile stress obtained at the fracture force, P_f:

$$S_f = \frac{1.5P_f\,(L_o - L_i)}{bh^2}$$

Various sources of internal and external errors in the testing process can affect the measured results of flexure tests. Errors classified as "internal" involve deviations from simple beam theory and involve test specimen geometry and properties.[5] A test specimen with an initial curvature resulting from residual stresses

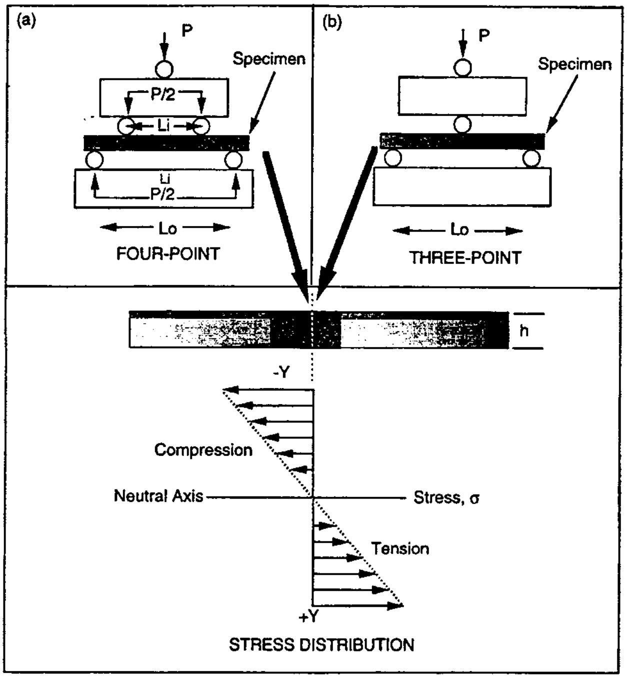

Fig. 2 Three- and four-point flexure testing and resulting stress state. (From Ref. 3 by courtesy of Marcel Dekker Inc.)

generated during machining would cause an internal error. Another example of an internal error would be excessive specimen deflection during testing. Support point frictional forces at large deflections will have a component aligned with the applied force that will increase the applied moment. "External" errors are those classified as being caused by incorrect test fixture geometry. Improper location of the inner load points is an example that causes external error. Another example is the generation of torque on the test specimen. This can be caused by an initially twisted test specimen, unparallel line loads, or nonuniform line loads at the contact points.[5,6] External error can also occur from compressive contact stresses at the support pins, which can result in localized crushing. This error can be reduced by using support pins above a critical radius r_c.[3]

To minimize the errors associated with flexure testing, the test specimen and test fixtures must adhere to certain restraints and standards. Test specimen geometry has been standardized based on error considerations. A common geometry in the United States is 3 by 4 by 50 mm for the test specimen and inner and outer spans of 20 and 40 mm, respectively, for the fixtures.[7] The test specimen must be isotropic and homogeneous to apply the maximum tensile stress equation previously given. In addition, the specimen should be as free as possible of surface defects as the maximum tensile stress occurs on the surface.

In comparing the three- and four-point flexure test methods, it is found that the four-point method is more appropriate for determining fracture strength because no shear stresses are generated as in the three-point test method. The three-point test method with its simpler geometry, however, may be more attractive for tests in which stable crack growth must be induced into the test specimen.[3]

Some of the flexure test standards that are applicable to advanced ceramics are listed here:

JISC, "Testing Method for Flexural Strength (Modulus or Rupture) of Fine Ceramics," R1601-1995

JISC, "Testing Method for Flexural Strength of Fin Ceramics at Elevated Temperature," R1604-1995

ASTM, "Standard Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperatures," C1161-94

ASTM, "Standard Test Method for Flexural Strength of Advanced Ceramics at Elevated Temperatures," C1211-92

Comité Européen de Normalisation (CEN),* "Advanced Technical Ceramics—Monolithic Ceramics—Mechanical Properties at Room Temperature—Part 1: Determination of Flexural Strength," EN843—1:1995

CEN, "Advanced Technical Ceramics—Monolithic Ceramics—Thermomechanical Properties—Part 1: Determination of Flexural Strength at Elevated Temperature," ENV820—1:1993.

2.2 Creep

Similar to the fracture testing methods previously discussed, the three test methods used to determine creep characteristics in ceramics are the tensile, com-

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* Comité Européen de Normalisation, Brussels, Belgium.

pressive, and flexural tests. The primary differences in the test fixtures are that the gauge section must be heated to the desired elevated temperature and an alternate method of measuring deflection such as extensometers must be employed because of the increased temperature. Figure 3 shows a typical test setup for a flexure test that utilizes extensometers.

Flexure Creep

Flexure testing using four-point loading yields the maximum tensile stress of a beam in bending given in the fracture strength section:

$$S_f = \frac{1.5P_f\,(L_o - L_i)}{bh^2}$$

In addition, the maximum tensile strain equation is given as:

$$\varepsilon_{\text{max}} = \frac{4h\delta_{\text{center}}}{(L_i)^2}$$

Compared to the tensile and compressive creep tests, flexure testing is less expensive, easier to design, and requires less test specimen preparation. However, the nonuniform stress state induced in the flexure testing results in difficulty in determining the creep characteristics of a material if it deforms differently in tension than in compression. This difference in tensile and compressive creep behavior is a result of cavitation among other factors. Conducting an additional test in either compression or tension allows for the determination of uniform stress-state creep characteristics from the nonuniform flexure test results. Additional errors from twisting, friction between test specimen and load points, and excessive contact stress at support points are also considerations.

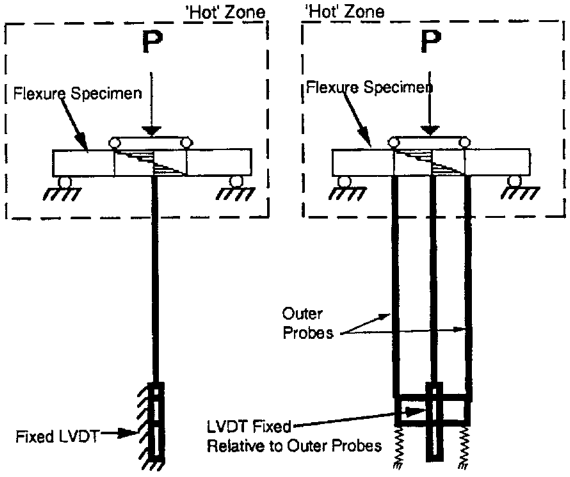

Fig. 3 Schematic of typical test setup for creep testing in flexure. (From Ref. 3 by courtesy of Marcel Dekker Inc.)

Tensile Creep

Tensile testing to determine creep characteristics yields a uniform stress state characterized by the equation:

$$\sigma_{\text{gs}} = \frac{P}{A}$$

The uniform stress state eliminates the problem of different deformations in tension and compression. This results in the much simpler calculation of stress and strain than with flexure testing. The test setup, however, is more expensive and difficult to implement. The test specimens required for creep tensile testing are also more difficult and time intensive to design and manufacture. In addition to stress gradients from the test specimen geometry, temperature differences induce an unwanted temperature gradient in the specimen from the grip contact portion of the specimen to the heated gauge section.[3] This temperature gradient becomes larger and poses more of a problem when cold grips are used. Unwanted failure in the grip interface portion of the specimen can also be a problem. Various test standards are applicable to tensile testing of advanced ceramics. ASTM C1291-95 directly applies to tensile testing for creep behaviors:

ASTM, ‘‘Standard Test Method for Elevated Temperature Tensile Creep Strain, Creep Strain Rate, and Creep Time to Failure for Advanced Monolithic Ceramics,’’ C1291-95

ASTM, ‘‘Standard Test Method for Tensile Strength of Monolithic Advanced Ceramics at Elevated Temperatures,’’ C1366-95

ASTM, ‘‘Standard Test Method for Monotonic Tensile Strength Testing of Continuous Fibre-Reinforced Advanced Ceramics with Solid Rectangular Cross-Sections at Elevated Temperatures,’’ C1359-96

Compressive Creep

Compressive testing to determine creep behavior, like tensile testing, results in a uniform stress state that allows for easy determination of creep characteristics. It has been shown that creep in compression can vary significantly from creep in tension so it is of value to conduct both tests to ‘‘get the best picture’’ of a material’s creep properties. Some advantages to the compressive test are the low cost in preparing the material as well as the small geometry of the specimen, which allows for more testing per sample material size.

There are some drawbacks to the compressive test as well. The test specimen must be aligned to a greater accuracy than tension or flexure tests to prevent unwanted bending and buckling. End constraints on the test specimen can result in shear stresses if the specimen ends are constrained, which results in a nonuniform stress state. In addition, temperature gradients can have an adverse effect on test result accuracy.

2.3 Hardness

Hardness is an important property to quantify in ceramics. Measured hardness indicates the ability of the ceramic to resist deformation by a hard object. Usually, Knoop or Vickers diamond indenters are used in conjunction with a microindentation hardness machine.[8] Rarely are the popular Rockwell and Brinell indenters used for ceramics research. Vickers indenters are used to characterize roughly 60% of the ceramic hardness values that are published.[8]

The indentation force should always be included with the hardness value. For the most accurate results, the entire force versus hardness curve should be measured as shown in Fig. 4.

Discrepancies can arise at different indentation forces. At higher forces, cracking can complicate the measuring process or make measuring impossible.[8] Measuring the hardness from the indentation, especially at small forces, is also a significant source of error in hardness testing. The hardness value can change based on the force value applied to the test specimen at small forces. Volume 8 of the *ASM Handbook*[8] recommends forces greater than or equal to 9.8 N for Vickers and Knoop indentations. Errors in the measurement of the indentation diagonal length essentially double the hardness error as the hardness value is proportional to the square of the diagonal length. A Versailles Advanced Materials and Standards (VAMS) round-robin test project conducted on alumina ceramic samples resulted in uncertainty in the hardness values given by the laboratories involved.[9] Although using numerous indentations can reduce some of this uncertainty, engineers and scientists conducting hardness tests should nevertheless keep this uncertainty in mind when considering their data.

Standards for measuring hardness for ceramics are listed below:

Vickers Hardness

ASTM, "Standard Test Method for Vickers Indentation Hardness of Advanced Ceramics," C1427-97

CEN, "Advanced Technical Ceramics—Monolithic Ceramics—Mechanical Properties at Room Temperature—Part 4: Determination of Vickers, Knoop and Rockwell Superficial Hardness Tests," prEN834-4

JISC, "Testing Method for Vickers Hardness of High Performance Ceramics," R1610:1991

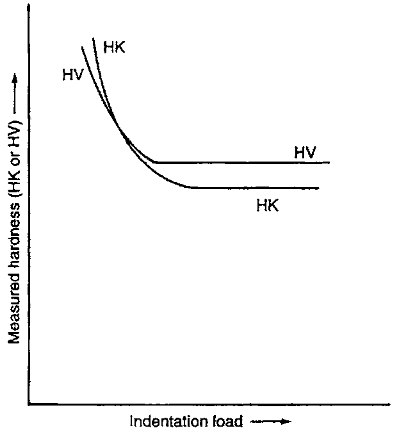

Fig. 4 Typical hardness versus load (force) plot. (From Ref. 8 *ASM Handbook, Vol. 8: Mechanical Testing and Evaluation,* ASM International, Materials Park, OH 44073-0002, Fig. 3, page 245.)

ISO, "Fine Ceramics (Advanced Ceramics, Advanced Technical Ceramics)—Test Method for Hardness of Monolithic Ceramics at Room Temperature," ISO 14705:2000

Knoop Hardness

ASTM, "Standard Test Method for Knoop Indentation Hardness of Advanced Ceramics," C1426-99

CEN, "Advanced Technical Ceramics—Monolithic Ceramics—Mechanical Properties at Room Temperature—Part 4: Determination of Vickers, Knoop and Rockwell Superficial Hardness Tests," prEN834-4

ISO, "Fine Ceramics (Advanced Ceramics, Advanced Technical Ceramics)—Test Method for Hardness of Monolithic Ceramics at Room Temperature," ISO 14705:2000

ISO, "Glass and Glass-Ceramics—Knoop Hardness Test," ISO 9385-1990.

2.4 Fracture Toughness

The brittle nature of ceramics results in low resistance to fracture, quantificial as fracture toughness, which is an important factor in many applications of ceramics. Fracture toughness is a measure of a specimen's ability to resist further growth of a crack. Low fracture toughness values increase the risk of catastrophic failure of a ceramic component. Ceramic matrix composites (CMCs) have better fracture toughness compared to monolithic ceramics as the additional reinforcing elements help to deter crack growth. The concepts of the linear-elastic fracture mechanics (LEFM) method commonly used for other materials can be applied to monolithic ceramics for the purpose of analysis. For ceramic matrix composites, ongoing research is being conducted regarding the application of the concepts of elastic-plastic fracture mechanics (EPFM) methods.[10]

There are various testing methods and setups for fracture toughness testing. Some methods included in the testing standard, ASTM C1421, are the single-edge precracked beam (SEPB) method, the surface crack in flexure (SCF) method, and the chevron notched beam (CNB) method.

The SEPB, SCF, and CNB methods all consist of a flexural test of a beam in which a short straight crack is induced into the tensile side. The primary differences are the methods by which the beam is deformed to introduce the preliminary short crack. All three methods have good calibration characteristics but have some drawbacks as well. With the SEPB method, it can be difficult to obtain straight-fronted cracks. Crack initiation in CNB testing can be difficult as a result of residual stresses from machining. SCF testing can only be performed on materials that produce high-quality cracks from the indentation.[10]

Standards organizations have introduced fracture toughness testing standards utilizing various testing methods. For example, ASTM C1421 generally uses a flexure test of a cracked bend bar while JIS R1607 utilizes only two test methods, SEPB and IF (indentation fracture). Standards that apply directly to fracture toughness testing of ceramics are given below.

Japanese Industrial Standards Committee, "Testing Methods for the Fracture Toughness of High Performance Ceramics," JIS R1607-1990

American Society for Testing Materials, "Standard Test Methods for the Determination of Fracture Toughness of Advanced Ceramics at Ambient Temperature," ASTM C1421-99

International Organization for Standardization, "Fine Ceramics (Advanced Ceramics, Advanced Technical Ceramics)—Test Method for Fracture Toughness of Monolithic Ceramics at Room Temperature by Single Edge Pre-Cracked Beam (SEPB) Method," ISO DIS15732 (in 1999)

2.5 High Strain Rate

Split Hopkinson pressure bar (SHPB) testing has primarily been used in the past to measure the plastic properties of metals. The fact that ceramics are brittle and many ceramic compounds and alloys show only elastic strains before fracture makes accurate testing and measurement more difficult than with more ductile materials such as metals.

A traditional SHPB test configuration is shown in Fig. 5.[11] The test setup consists of an incident bar, a transmission bar, and a striker bar. The ceramic test specimen is placed in between the incident and transmission bars as shown. The striker bar strikes the incident bar at a predetermined velocity by being launched from a gas gun. When it impacts the incident bar, it generates a compressive pulse that continues to travel to the test specimen. Part of the incident pulse travels through the test specimen, and the remaining part is reflected back into the incident bar. The stress pulses are measured by strain gauges placed at midpoints on the transmission and incident bars. An oscilloscope displays the measured pulses. The stress, strain, and strain rate equations are given as[12]:

$$\sigma_s(t) = \frac{A_o E_o}{A_s}\, \varepsilon_T(t)$$

$$\dot{\varepsilon}_s = -\frac{2c_o}{l_s}\, \varepsilon_R(t)$$

$$\varepsilon_s(t) = \int_0^t \dot{\varepsilon}_s(t)\, dt$$

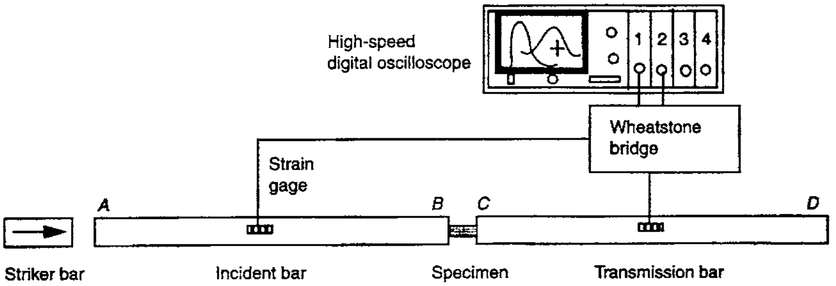

Fig. 5 Test setup schematic for traditional split Hopkinson pressure bar testing. (From Ref. 11 *ASM Handbook, Vol. 8: Mechanical Testing and Evaluation,* ASM International, Materials Park, OH 44073-0002, Fig. 1, page 497.)

where E is Young's modulus, l is length, A is cross-sectional area, σ is stress, ε is strain, $\dot{\varepsilon}$ is strain rate, and t is time. Subscripts T, s, o, and R refer to the transmitted pulse, specimen, bar, and reflected pulse.

Some of the inherent properties of ceramics such as brittleness and high strength, conflict with assumptions that are made in deriving the stress, strain, and strain rate equations.[11] An example is the assumption that the bar end surfaces remain flat and parallel during the deformation of the test specimen. The hard nature of ceramics can cause the test specimen to indent into the steel bar ends, resulting in stress concentration around the test specimen end faces. These stress concentrations result in nonuniform, nonuniaxial stress state in the test specimen, which violates another assumption in using the equations, which is that the stress state is uniform and uniaxial. The stress concentrations can consequently lead to chipping and failure of the ceramic test specimen through microcrack initiation.

One way to minimize the problem of test specimen damage is to place tungsten–carbide inserts in between the test specimen and bar ends as shown in Fig. 6.[11] The high strength of the inserts prevents the ceramic test specimen from indenting into the incident and transmission bars, thereby reducing stress concentrations. However, the tungsten–carbide inserts can have the adverse effect of altering the incident, transmitted, and reflected stress-wave properties that can result in inaccurate strain gauge measurements. This can be prevented by selecting tungsten–carbide inserts with the same impedance as the bar material. Selection of the diameter of the inserts can also help solve this problem. Typically, the insert lengths are one fourth of the test specimen length.[11]

2.6 Fatigue

Fatigue testing is an important design tool for the designer of ceramic components where reliability and lifetime estimates need to be made. Fatigue tests for ceramics generally cover the three situations of cyclic fatigue, static fatigue, and dynamic fatigue.[13] Cyclic fatigue is the periodic loading of a component under various load ratios usually denoted as R:

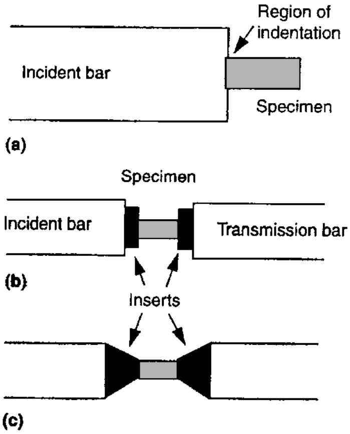

Fig. 6 Incident bar and transmission bar protection using tungsten–carbide inserts. (From Ref. 11 *ASM Handbook, Vol. 8: Mechanical Testing and Evaluation,* ASM International, Materials Park, OH 44073-0002, Fig. 3, page 499.)

$$R = \frac{\sigma_{\text{min}}}{\sigma_{\text{max}}}$$

Common scenarios for the stress ratio include $R > 0$ (both minimum and maximum stresses in tension or compression) and $R < 0$ (minimum stress in compression and maximum stress in tension). The simple loading scenario would involve a sinusoidal loading that varied continuously with a minimum and maximum stress. In real-world examples, however, the loading can be much more complicated.

Static fatigue testing involves slow crack growth of a test specimen with a constant tensile force under the desired conditions. The length of time for the test specimen to fail and the applied stress are measured and used to obtain the fatigue characteristics by using various fatigue equations. Dynamic fatigue testing involves applying a constant, increasing stress (i.e., constant nonzero stress rate) to a test specimen under desired conditions. The stress rate and maximum applied stress at failure are measured and used in conjunction with various fatigue equations to determine the fatigue characteristics.

Fatigue testing can further be divided into the two categories of "direct" and "indirect." Direct methods, also known as fracture mechanics methods, involve running tests with test specimens with previously induced cracks and directly observing crack growth. Indirect methods, also known as strength measurement techniques, involve measuring the strength of the test specimen over time intervals and using the data to estimate fatigue properties.

Indirect Methods

Static loading utilizing the indirect method involves subjecting a tensile or flexure test specimen to a constant load under the desired environment conditions. The applied force stress and the amount of time for specimen failure are measured and used to estimate the fatigue properties of the ceramic material. A typical plot of failure stress versus time to failure is shown in Fig. 7.

Uniaxial flexural tests in three- or four-point loading situations are performed under the directions given in JIS R1601, "Test Method for Flexural Strength (Modulus of Rupture) of Fine Ceramics." At least three levels of stress are

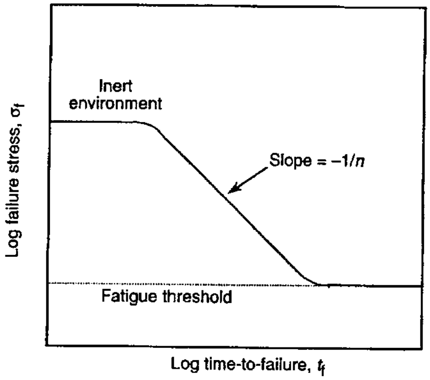

Fig. 7 Failure stress versus time to failure. (From Ref. 14 *ASM Handbook, Vol. 8: Mechanical Testing and Evaluation,* ASM International, Materials Park, OH 44073-0002.)

recommended. The fatigue strength can be related to the failure time with the following equation[14]:

$$t_f = \frac{B}{\sigma_f^2}\left[\left(\frac{\sigma_i}{\sigma_f}\right)^{n-2} - 1\right]$$

where B is a variable determined from crack geometry, fracture toughness, loading situation, and K and n, which are material and environment constants; σ_i is the inert strength at which no crack extension occurs for noncorrosive conditions.

Indirect dynamic loading usually involves tensile or flexure test specimens subjected to a constant stress rate. The applied stress rate and the failure stress are measured and used to estimate fatigue properties. Figure 8 shows a typical plot of failure stress versus the stress rate.

The standard, ASTM C1368, gives guidelines for fatigue strength estimating via dynamic loading. It involves four-point flexure testing of a ceramic specimen in accordance with ASTM C1161 "Standard Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature." The fatigue strength and applied stress rate are related by the equation[14]:

$$\sigma_f = [B(n + 1)\sigma_i^{n-2}\dot{\sigma}]^{1/n+1}$$

where the same variable definitions apply as in the static loading equation.

There are various methods for applying periodic loads to ceramics. With bending fatigue tests, a periodic force with a specified frequency and stress ratio is applied to three- or four-point flexure test setups. Cyclic tensile fatigue tests involve cyclically loading a test specimen with a testing apparatus with specified load ratio and frequency. Various test specimen shapes can be used and tension–compression cyclic fatigue (stress ratio < 0) has been performed utilizing a button-head specimen and clamping fixture. Rotary bending is also used for fatigue testing ceramics and is especially useful for analyzing components such as shafts that undergo similar stress states while in use.

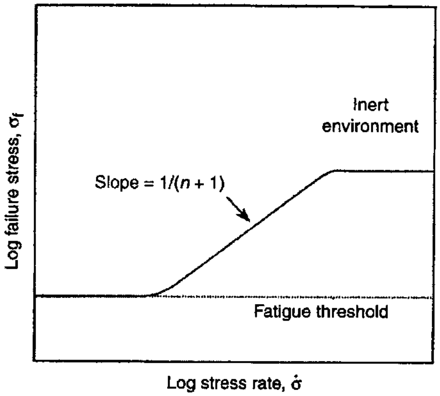

Fig. 8 Failure stress versus stress rate. (From Ref. 14 *ASM Handbook, Vol. 8: Mechanical Testing and Evaluation,* ASM International, Materials Park, OH 44073-0002.)

Direct Methods

Direct methods, also known as fracture mechanics methods, use fatigue tests that employ test specimens with an induced crack.[14] Crack growth is observed directly or via strain gauges or other measuring devices.

One testing technique is the double-cantilever beam method, shown schematically in Fig. 9. For the configuration shown, the stress-intensity factor and fracture toughness can be found utilizing the equation[14]

$$K_I = \frac{Pa}{h^{3/2}\sqrt{Bb}}\left(3.47 + 2.32\,\frac{h}{a}\right)$$

where B is the thickness of the test specimen, b is the web thickness, a is the crack length, h is half the specimen height, and P is the applied force. Some standards that directly apply to fatigue testing of ceramics are given below.

- Japanese Industrial Standards Committee, "Test Methods for Static Bending Fatigue of Fine Ceramics," JIS R1632—1998
- Japanese Industrial Standards Committee, "Test Method for Flexural Strength," JIS R1601—1995
- American Society for Testing Materials, "Standard Test Method for Determination of Slow Crack Growth Parameters of Advanced Ceramics by Constant Stress-Rate Flexural Testing at Ambient Temperature," ASTM C1368-99
- American Society for Testing Materials, "Standard Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature," ASTM C1161-99
- Japanese Industrial Standards Committee, "Test Method for Bending Fatigue of Fine Ceramics," JIS R1621—1996
- American Society for Testing Materials, "Standard Test Method for Flexural Strength of Advanced Ceramics at Elevated Temperatures," ASTM C1211-95

3 THERMAL TESTING

3.1 Thermal Expansion

Thermal expansion is an important property that quantifies the volume change a material undergoes when it is subjected to temperature changes. Typically,

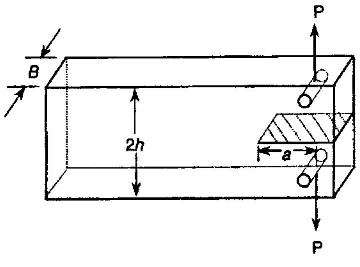

Fig. 9 Double-cantilever beam test method. (From Ref. 14 *ASM Handbook, Vol. 8: Mechanical Testing and Evaluation,* ASM International, Materials Park, OH 44073-0002.)

materials expand when heated and contract when cooled. This characteristic is valuable to the engineer or scientist involved in ceramic design or research. The coefficient of thermal expansion (COTE), α, is strongly related to the strength of the atomic bonds. Energy must be put into the material for the atoms to move from their equilibrium positions. Typically, ceramics, which have strong ionic or covalent bonds, have lower thermal expansion coefficients than metals. The average COTE is simply the change in the length of the material per unit length per unit temperature:

$$\alpha = \frac{\Delta L}{L_o \Delta T}$$

The volume COTE, α_v, is the change in volume of the material for a given temperature. Consequently, for an isotropic material the volume COT is given as:

$$\alpha_v = 3\alpha$$

Thermal expansion is a tensor property that is different along individual crystallographic axes.[15] This varying expansion can cause residual stresses in some test specimens that can actually cause cracks between the crystal faces in some rare situations. This thermal cycling cracking will manifest itself in the form of a hysteresis curve from expansion data during thermal cycling tests.

Typically, the COTE is lower for materials having a high melting point. The linear COTE is a more exact definition and continuously changes with regard to temperature. The COTE for materials is usually listed in handbooks as a complicated temperature-dependent function or is shown as a constant that is valid only for a specified temperature range. Taking into account the temperature dependence of the linear COTE, experimental measuring gives the more exact equation[15]

$$\alpha(T_i) = \left(\frac{\partial L(T)}{\partial T}\right)_{T=T_i} \frac{1}{L(T)}$$

where $L(T_i)$ is the experimentally measured length of the specimen at a given temperature T_i. The difference in the linear COTE and the average COTE is shown graphically in Fig. 10.

Understanding the thermal expansion of a given ceramic is especially important in design applications such as composites that combine structurally distinct constituents (fiber, matrix, etc.) of different materials (ceramic, metal, etc.) with different COTE. For example, a ceramic with a low COTE combined with a metal possessing a high COTE can result in critical stresses for a given temperature change.

Dilatometry

The most popular method for measuring thermal expansion is dilatometry.[15] The test setup is simple, consisting of a cylindrical test specimen placed between a

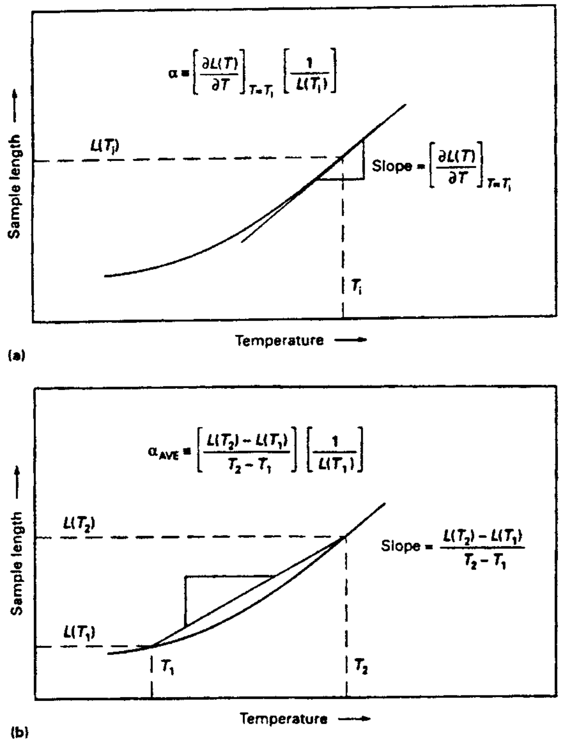

Fig. 10 Difference in linear COTE and average COTE. (From Ref. 15 *Engineered Materials Handbook, Vol. 4: Ceramics and Glasses,* ASM International, Materials Park, OH 44073-0002, Fig. 1, page 612.)

fixed base and a movable push rod. The ceramic test specimen is heated at a fixed rate, which displaces the push rod a given distance that is recorded by a sensing device. Software is readily available to record the data and determine the temperature-dependent COTE and the average COTE record.

Dilatometers can be of the single or double push rod variety. The single push rod method involves first calibrating the test apparatus in terms of the change in length versus temperature-sensing device with a test specimen possessing a known COTE. Next, the ceramic test specimen is tested compared to the previously measured standard to determine its COTE. Calibrating the test apparatus with a standard reference specimen first in this manner can correct for problems such as dilatometer material expansion, thermal gradients, and nonlinearity in the heating rate.[15] The double push rod design tests the ceramic test specimen and the standard reference material side by side. The thermal expansion data is then calibrated by comparing it with the standard reference material with a known COTE.

Other Techniques

X-ray diffraction can give insight into the structure of ceramics. The tensor components of thermal expansion can be determined with X-ray diffraction through the measuring of changes of the interplanar spacing with relation to the temperature. One distinct advantage with this method is that only a very small sample of the ceramic is required. Another advantage is that measuring the tensor components of thermal expansion of a specimen is possible with X-ray diffraction. This can be important when trying to estimate residual stresses that will occur during thermal cycling.

Interferometry is a technique that involves placing the test specimen between two reflecting surfaces and measuring the displacement through the movement of the surfaces. The basis for this being that parallel reflective surfaces a short distance apart will show interference fringes when illuminated by monochromatic light. As the reflective surfaces move apart from the specimen expansion, the interference fringes move past a reference point on one of the reflecting planes. The expansion of the sample in terms of length can be expressed as[15]:

$$\frac{\Delta L}{L} = \frac{\lambda N}{2L} + \frac{A}{L}$$

where N is the number of interference fringes that pass the reference point, L is the specimen length, ΔL is the change in the specimen length, λ is the wavelength of the light source, and A is the correction for the light source based on the atmosphere in the measuring environment. Restrictions on the test specimen geometry make this technique less popular than dilatometry methods. One advantage to this technique, however, is that ceramics with volatile components can be tested by interferometry inside a closed, heated test chamber where the volatile component's vapor pressure can be controlled.

3.2 Thermal Conductivity

Thermal conductivity is a measure of the rate of heat transfer in a given material by conduction. The heat flowrate through a material is proportional to the heated area of the material and the temperature gradient across the specimen. This proportionality yields the coefficient of thermal conductivity, κ, which is shown in the following equation[16]:

$$\frac{dQ}{dt} = -\kappa A \frac{dT}{dx}$$

The negative sign on the right side indicates that heat flows from a higher to a lower temperature. The rate of heat flow is dQ/dt, A is the cross-sectional area of the material, and dT/dx is the temperature gradient.

Two important mechanisms involved in transferring thermal energy are lattice vibrations (or phonons) and transfer of free electrons. Valence electrons travel from hot to cold areas with the energy gained, and then transfer their energy to other atoms. Many electrons in ceramics cannot be excited into the conduction band except at quite high temperatures because the energy gap is typically too large.[16] Consequently, heat transfer in ceramics is primarily a result of lattice vibrations. This results in ceramics typically having a much lower thermal conductivity than metals. However, not all ceramics have low thermal conductivity. Advanced ceramics such as AlN and SiC have good thermal conductivity and low electrical conductivity. Consequently, they are good for electronic applications where dissipating heat is a requirement. Thermal conductivity is an important aspect of materials for microelectronic substrates and electronic packaging materials. The development of higher density circuits makes heat dissipation an increasingly difficult problem to overcome.

Guarded Hot Plate

The guarded hot plate technique is a comparative method that measures thermal conductivity through the application of thermocouples to a cylindrical ceramic test specimen sandwiched between two cylindrical sections of a reference material with the same diameter and with a known thermal conductivity. The test setup is shown in Fig. 11. Thermally conductive paste is applied to the mating surfaces to ensure adequate heat transfer between materials. Using a reference material with a thermal conductivity similar to that of the ceramic test specimen will yield the best results. Stack heaters at the top and bottom of the cylinder stack introduce heat into the materials. The thermocouples are positioned near the mating surfaces on the reference and test cylinders. The thermocouples determine the temperature gradient and then the steady-state heat flux is established between the stack heaters. Using the thermal conductivity of the reference material and the measured temperature gradients in the two test specimen cylinders sandwiching the reference cylinder, the heat flux through the entire stack can be calculated using the thermal expansion equation previously given. These two independent calculations of heat flux through the stack are then averaged, and the average is used as the heat flux value for the ceramic test specimen. In addition, the difference in the two calculated values give the heat loss value for the stack. The coefficient of thermal conductivity, κ, can then be determined

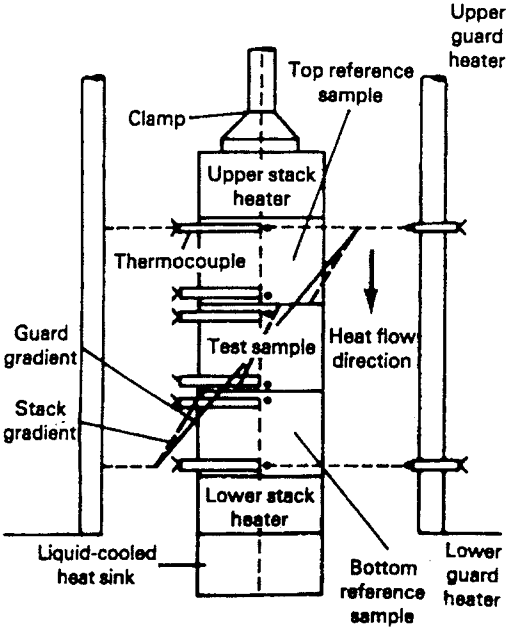

Fig. 11 Guarded hot plate test setup. (From Ref. 15 *Engineered Materials Handbook, Vol. 4: Ceramics and Glasses,* ASM International, Materials Park, OH 44073-0002, Fig. 2, page 613.)

because the average heat flux through the stack, the area of the test specimen cylinder, and the measured temperature gradient along the ceramic test specimen's length are all already known. To eliminate convective heat losses, measurements can be made in a vacuum or reduced atmosphere environment.

Laser Flash Method

Another method for determining thermal conductivity is the laser flash method, which involves quickly heating a thin ceramic specimen on one side via a quick "thermal pulse" from a laser and then using the measured temperature values over time of the back face of the specimen to calculate thermal diffusivity and conductivity. Thermal diffusivity, α, is a different means of expressing a material's heat conduction properties. Thermal diffusivity takes into account that heat can diffuse through a material subject to different boundary conditions, causing both spatial and temporal variations of temperature.[16] Thermal diffusivity is a temperature-dependent material tensor property that is defined by the equation

$$\frac{dT}{dt} = \alpha_{ij}(T)\nabla^2 T$$

The relationship between thermal diffusivity and thermal conductivity is given by the equation

$$\alpha_{ij}(T) = \frac{k_{ij}(T)}{\rho(T)c_p(T)}$$

where $\rho(T)$ is the temperature-dependent density and $c_p(T)$ is the specific heat capacity of the test specimen.

The ceramic test specimen is in the shape of a thin disk and is at a constant temperature T, when the laser flash exerts a thermal pulse on the front face. Figure 12 shows a schematic of the test setup.

The assumptions here are that the thermal energy is deposited uniformly over the front face of the test specimen, that the heat travels along the thickness of the specimen only to the back face, and that the pulse heats the sample only.[16] Then, knowing the temperature of the back face at times after the pulse is introduced into the test specimen, the thermal diffusivity of the test specimen

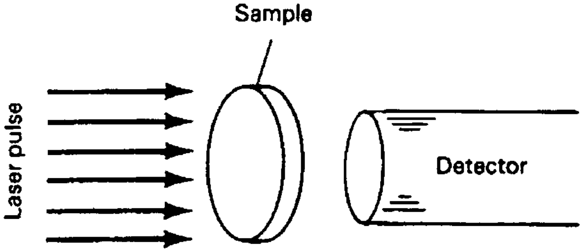

Fig. 12 Schematic of laser flash method test setup. (From Ref. 15 *Engineered Materials Handbook, Vol. 4: Ceramics and Glasses,* ASM International, Materials Park, OH 44073-0002, Fig. 3, page 614.)

can be calculated using various algorithms. Different available algorithms use various temperatures of the back face to calculate thermal diffusivity.

An advantage of the laser flash method is that, in addition to having a simple test setup, it can be used over a large range of thermal conductivities and ceramic specimen temperatures. Also, it is quite popular for measuring thermal conductivities at high temperatures as maintaining a steady-state condition is difficult at higher temperatures.

Other thermal pulse methods using different sources such as electron beams or quartz flash lamps are also possible utilizing very similar test procedures.

3.3 Heat Capacity

Heat capacity is a property that refers to the amount of energy that must be added or subtracted from a material to raise or lower its temperature. The amount of energy required to raise the temperature of a material by a degree varies from material to material based on its properties. Specific heat at a constant pressure, c_p, is the most common expression of a material's heat capacity and is defined as the amount of heat needed to raise the temperature of one gram of a substance by one kelvin at a constant pressure. The specific heat is derived from the enthalpy, H, and this relation is shown in the following equation:

$$c_p = \left(\frac{\partial H}{\partial T}\right)_p$$

where T is the temperature and the subscript p indicates constant pressure. From this equation, we can see that the specific heat is the slope of the enthalpy versus temperature change. The specific heat value can be considered a constant for a short range of temperature, but the actual measured specific heat term is expressed as a polynomial to express the nonlinearity of the specific heat with the temperature. This more defined specific heat capacity and its form are given in the equation

$$C_p(T) = a + bT + cT^2 - dT^{-2} + eT^{-1/2}$$

However, the form of equation above does not provide a specific heat for all temperature ranges of many materials. Various equations of this form must be determined for various temperature ranges. There are various methods for determining the heat capacity of ceramics.

Calorimetry

Calorimetry can be used to determine the heat capacity of a ceramic material. A specific amount of a material test specimen is heated to an initial temperature with an external furnace and then deposited into a calorimeter of a lower temperature. The calorimeter measures the heat energy that the test specimen gives off while cooling to the equilibrium temperature that is between the specimen and calorimeter temperature. Measurements of the enthalpy, H, at various temperatures gives a plot of $H(T)$ versus T. Consequently, the specific heat, c_p, can be calculated from this data.

An advantage of calorimetry is that it can calculate specific heats over a wide temperature range. A drawback, however, is that the method is insensitive to transitions with minor changes in enthalpy.[17]

Differential Scanning Calorimetry

Differential scanning calorimetry is the most popular method for measuring the specific heat of a ceramic. This type of calorimeter measures the heat flowrate to a ceramic test specimen while it is heated at a given constant rate. A computer monitors the temperature of the test specimen and makes adjustments to keep the temperature of the ceramic test specimen increasing at a constant rate. Consequently, the specific heat, c_p, of a ceramic test specimen of known mass m can be calculated from the following equation[17]:

$$\frac{dH(T)}{dt} = mc_p(T)\,\frac{dT}{dt}$$

This method is commonly used and there are various commercial differential scanning calorimeters available. One of the major drawbacks to this technique is that commercial setups can only be used up to a temperature of about 800°C.[17] In addition, testing errors resulting from thermal resistance between the test specimen and heat flow sensing device and other sources requires the use of a comparative process when testing. A different material with a known heat capacity is tested under the same conditions and any difference is noted and incorporated into the error evaluation of the ceramic test specimen being investigated.

4 NONDESTRUCTIVE EVALUATION TESTING

Many nondestructive evaluation (NDE) testing techniques are commonly used for evaluating metals and other materials. Applying these techniques, however, to ceramics does not always provide adequate results because of the unique nature of ceramics that impede many of the typical testing techniques. The increasing use of advanced ceramic materials in critical applications along with properties that make them sensitive to quite small defects increases the need to define NDE testing methods directly applicable to ceramics.

Advanced ceramics are typically brittle in nature. Defects as small as 10 μm can be critically detrimental and must be avoided. They can be prevented with careful process control of the fabrication of the ceramic materials from fine powders or with careful inspection of the finished ceramic parts. Both strategies can be applied with NDE testing as a major component.

4.1 Ultrasonography

Ultrasonic testing is a common NDE testing technique that can detect and describe flaws and material conditions that other methods are unable to do. Ultrasonic waves are propagated through the material and the waves are disrupted at the discontinuities in the material such as defects, voids, or cracks. The waves are scattered or partially reflected at these discontinuities and from this action, a measure of the discontinuity characteristics such as location, size, and shape are revealed. The ultrasonic method does not always reveal everything about the discontinuity to the degree desired but is still an invaluable tool.

The distinguishing characteristic of ceramics that make typical ultrasonic testing techniques less successful are the high ultrasonic velocities and the smaller critical defect size that must be detected. To detect and characterize small defects, the equipment must be modified so that the ultrasonic wave frequency is increased. An ultrasonic wave with a wavelength of similar size to the defect is required to detect the defect. Consequently, with a ceramic material where a critical defect can be a very small value such as 10 μm, a wavelength of approximately the same size is needed.[18] This indicates a very high frequency, f, is required based on the frequency equation

$$f = \frac{v}{\lambda}$$

where λ is the wavelength and v is the longitudinal wave velocity.

An important property to be discerned in a ceramic material is its bulk porosity. Ultrasonic testing can reveal the material's bulk porosity even though the individual pore size is much smaller than the investigating wavelength. The porosity can be characterized by measuring the ultrasonic velocity and attenuation. Measuring the ultrasonic velocity typically entails using a transducer to a face of a ceramic specimen with parallel faces and a known thickness. The round-trip travel time of the acoustic pulse is measured with the transducer in a pulse-echo mode. The ultrasonic velocity v is given by the equation

$$v = \frac{2d}{t}$$

where d is the thickness of the test specimen and t is the time for round-trip travel. It has been found that there is a linear relationship between ultrasonic velocity and porosity. Once the ultrasonic velocity has been measured, the variations in porosity in a specimen can be shown with the aid of ultrasonic C-scans.

Ultrasonic attenuation is the process by which the ultrasonic beam, when propagating through the test specimen, loses energy. Grain boundaries, pores, voids, and other internal defects cause scattering of the waves, resulting in a lower energy beam. This attenuation in the ultrasonic beam is expressed by the equation

$$I = I_0 \exp(-\alpha x)$$

where I is the beam intensity, x is the distance into the test specimen ($x = 0$ at surface), and α is the ultrasonic attenuation coefficient of the material. Once the attenuation of a material is characterized, various imaging equipment can map the amplitude image of a specimen, which visually describes the porosity of a test specimen.

A standard that applies directly to ultrasonic testing is ASTM E494, "Standard Practice for Measuring Ultrasonic Velocity in Materials."

4.2 Radiography

Radiography uses radiation to characterize a material's structure by examining the interaction between the electromagnetic wave and the material itself. The detection of voids, cracks, pores, and other defects is the primary goal of this technique. The detection of these defects is a result of the attenuation and scatter of the radiation as it passes through the material. Various radiation sources can be used in the application of radiography but the most versatile has been found to be X-ray sources. There are two common methods for employing X-ray sources to discover discontinuities and defects in a material's structure. One method is to create a two-dimensional image of the test specimen or component where the variation of the image intensity indicates the degree of attenuation. Another approach is to use many attenuation image "slices" and an algorithm to develop a three-dimensional image.

X-ray microradiography techniques employ a divergent X-ray beam to produce a two-dimensional image of the test specimen or component being investigated. High contrast and clarity are required when trying to detect the inherently small defects in most ceramics. One common method of applying X-ray microradiography is using a contact method where the test specimen or component being studied contacts an imaging medium, which results in a high-resolution image. Another method uses X-ray sources that are typically less than 100 μm, which allows direct magnification of the component or test specimen image.[18] The ability of these methods to detect defects is dependent on the degree of contrast and resolution that is obtained in the image. One disadvantage to this two-dimensional technique is that cracks and other linear defects orientated transverse to the beam direction will be much more difficult to detect.

X-ray computed tomography utilizes many two-dimensional "slice" images of the X-ray attenuation to form a three-dimensional representation of the object being investigated. This is especially useful when trying discover defects and discontinuities in a complex three-dimensional component. It is used primarily as a research tool and a few of the main drawbacks to the technique are its high cost and complexity.

5 ELECTRICAL TESTING

Ceramic materials have important functions in various electrical and electronic applications. Ceramics, with their unique properties, provide capacitive, insulative, conductive, resistive, and other functions in electronic circuitry. An example of the important role ceramics have in electronics is the use of advanced ceramics such as alumina oxide (Al_2O_3) as the substrate material in electronic packaging. Increases in circuit density have consequently resulted in more stringent requirements being placed on substrate materials. A substrate is desired that has thermal expansion characteristics that closely match with silicon to prevent critical thermal stresses. A low dielectric constant is also needed to improve signal processing. In addition, thermal conductivity is a high priority to dissipate heat from the high-density circuit. Based on stringent requirements such as these, the development of new advanced ceramics and subsequent testing to quantify the development and selection of ceramic materials for important electrical/electronic applications is of increasing importance. Directly adapting current electrical testing techniques to ceramics serves to improve the process of ceramic integration into electronics and other electrical applications.

5.1 Electrical Resistance at Elevated Temperatures

An important aspect of ceramic use in electronic applications is the variation of electrical resistance as the temperature rises. With the trend toward higher and higher density circuits, characterizing substrate and other chip component properties at elevated temperatures is quite important.

Measuring electrical resistance at high temperature involves applying a voltage across a test specimen contained in a heating furnace. A ceramic test specimen is mounted between the electrodes in the furnace that has heated the test specimen to the desired temperature. A voltage of 500 V DC is applied across the test specimen for one minute and the volume resistance is measured. The process is repeated for various temperatures until maximum test temperature is reached. The volume resistivity, ρ, is then calculated from the following equation:

$$\rho = \frac{A}{h} R_v$$

where R_v is the measure volume resistance, A is the area of the electrode, and h is the average thickness of the ceramic specimen in the area covered by the electrode. Further specifics are given in ASTM D1829—90, ‘‘Electrical Resistance of Ceramic Materials at Elevated Temperatures.’’

5.2 Flexural Strength of Electronic-Grade Ceramics

Another important test for characterizing electronic ceramic components is the flexural strength test. Flexural strength testing is already standardized, but application to electronic-grade ceramics requires slight modification. The testing sample sizes are much smaller and commonly referred to as ‘‘microbars.’’ Changes in the size of the bending test fixtures, load application, and material preparation are also required. The specifics are outlined in ASTM F417—78, ‘‘Standard Test Method for Flexural Strength of Electronic-Grade Ceramics.’’ The flexural strength is still calculated using the standard equation:

$$S = \frac{3PL}{2bd^2}$$

where S is the flexural strength, P is the force at fracture, L is the distance between supports, and b and d are the width and thickness, respectively, of the specimen.

6 SUMMARY

Testing of ceramics has been standardized or formalized for mechanical, thermal, NAE, and electrical aspects of these brittle materials. Although testing for many properties and performance related to these aspects is accepted, methodologies for material selection and design with brittle materials are still being developed. As these methodologies mature and become widely accepted, additional test methods related to the required properties and performance will be proposed, developed, and approved.

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CHAPTER 24

NONDESTRUCTIVE INSPECTION

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1 INTRODUCTION

This chapter deals with the nondestructive inspection of materials, components, and structures. The term *nondestructive inspection* (NDI) or nondestructive evaluation (NDE) is defined as that class of physical and chemical tests that permit the detection and/or measurement of the significant properties or the detection of defects in materials, components, or structures without impairing their usefulness. The NDI process is often complicated by the fact that many modern materials are by nature anisotropic. Most of the current NDI techniques were developed for isotropic materials such as metals. The added complication due to the anisotropy usually means that an inspection is more complicated and requires more analysis than had been the case previously.

Inspection of complex materials or structures is often carried out by comparing the expected NDI data with a standard and noting unexpected deviations. This means a high-quality standard must be available for calibration of the inspection instrumentation. Furthermore, high-quality standards also must contain special implanted flaws that mimic those that naturally occur in these materials or structures. Without a high quality standard to test and calibrate the inspection process, the analysis of NDI results can be significantly in error. For example, to estimate the amount of porosity in a cast component from ultrasonic measurements, standards with calibrated levels of porosity must be available to calibrate the instrumentation. Without such standards, estimation of porosity from ultrasonic data is a highly speculative process.

There are many methods available to conduct the NDE or NDI. This chapter covers some of the important and some less well known NDI tests. Since information on the less often used tests is not generally in standard texts, additional sources of information are listed in the references at the end of the chapter.

Inspection instrumentation must possess four qualities to receive widespread acceptance in the NDI community. These qualities are

1. *Accuracy.* The instrument must accurately measure some property of the material or structure that can be used to infer either properties or the presence of flaws.
2. *Reliability.* The instrument must be highly reliable. That is, it must consistently detect and quantify flaws or a property with a high degree of reliability. If an instrument is not reliable, then it will misdiagnosed properties or flaws can lead to failure of the component, property damage, and potential loss of life.
3. *Simplicity.* The instruments that are most useful are those that may be used by factory or repair-level technicians. Instrumentation that requires highly skilled operators are very rarely used by the inspection community.
4. *Low Cost.* An instrument need not be low cost in an absolute sense. Instead, it must be inexpensive relative to the value of the component under test or the cost of a failure or aborted mission. For flight-critical aircraft components, as much as 12% of the value of the component may be spent in inspection.

While this list covers the desirable characteristics for instrumentation, those that apply to the inspection process are enumerated in the Section 8 on reliability.

1.1 Information on Inspection Methods

To the engineer or scientist confronted by a new inspection requirement there arises the question of where to find pertinent information regarding an inspection procedure and its interpretation. Fortunately there are many potential sources of information about instrumentation and techniques available for nondestructive inspection. A brief examination of the literature is presented next. Many of these references were generated as a result of the demands on materials used in aerospace structures. There was an attempt to provide traditional scientific and engineering books and journals that would be available in any well-stocked library. However, with the rise of the Internet there are now many electronic sources of information available on the World Wide Web. These include such information sources as: the library catalog, societal home pages, an on-line journal devoted to NDI, manufacturers of instrumentation with on-line demonstrations of services, inspection software, and on-line chat rooms devoted to solving inspection problems. The references include the most commonly used sources. However, with new electronic sources appearing daily this is only a brief snap-shot of the sources available at the beginning of the twenty-first century.

General NDE Reference Books

Some general references to the NDE techniques are given in Refs. 1–22. The reader will note that some of these citations are not recent; they were included because of their value to the engineer who does not possess formal training in NDE. Additionally, some older works were chosen because of the clarity of presentation, completeness, or their usefulness to the inspection of complex structures.

NDE Journals

The periodical literature is a source of the latest research results of new or modified inspection methodologies.[23–31] Some journals are no longer available, but are included because of their archival value or because the may contain data available nowhere else. Whenever possible, World Wide Web addresses are provided to give the readers ready access to the publisher.

1.2 Additional References

There are many potentially useful references for the reader interested in using or modifying NDE techniques.[4,7,11,13,32] For those new to the field the American Society for Testing and Materials (ASTM) standards is particularly valuable because it gives very detailed directions on many NDE techniques. More importantly, it is widely accepted as a standard reference for standard inspections. The other references are given for those situations where standard inspection methods are not sufficient to detect the material condition of interest.

1.3 Electronic References

There are many useful electronic references for those working in NDE technology. Of the many useful sites on the World Wide Web (www), only a few are included here. Many sites contain links to other sites that may contain information on a special topic of interest to the reader. Because the web is growing at a fantastic rate, the list in the bibliography represents a very brief snapshot of the information available to the NDE community. Even some useful www sites associated with government agencies were not included due to space limitations. The list of sites is divided into sections associated with NDE societies,[33–37] institutes[38–42] government agencies,[43–46] publishers,[29,45,47–49] and general-interest sites.[37,50–54]

1.4 Future NDE Capabilities

At this point the reader might be tempted to ask if there are new technologies on the horizon that will enable more cost-effective, anticipatory inspections, or monitoring of materials and structures? This answer to this is an emphatic yes. There are new developments in solid-state detectors that should significantly impact both the capability of inspection and its cost as well. For example, optical and X-ray detectors will give the inspector the ability to rapid scan large areas of structures for defects. By coupling this technology to computer algorithms that search image information, the inspection of large areas can be automated providing more accurate inspections with much less operator fatigue.

The area of data fusion is just being explored in the NDE field. This means that data collected with one technique can be combined with another technique to detect a range of flaws not detected with either independently. The data from several techniques that are coupled at the basic physics level provide a more complete description of the microstructural details of a material than is now possible.

Finally, with the rapid development of new silicon-based devices [microelectromechanical systems (MEMS)] that are implantable into a material at the time of manufacture offer real-time, health-monitoring capabilities. This new capability is permitting inspectors to detect and quantify material or structural degradation in time to prevent failure, thus avoiding unexpected down time. Many of these devices can be interrogation through a wireless radio frequency (RF) link. This reduces the burden of inspection while giving the inspector the ability to inspect areas of structure that are now termed ‘‘hidden.’’ For more information about this rapidly evolving area the reader is referred to the literature for more detailed data.[29,54–62]

This chapter is just a brief review of the more commonly used NDI methods. Table 1 provides a brief review of the common NDI methods, types of flaws that each detects, and their advantages and disadvantages. Those who require more detailed information on any method should consult one or more of the references at the end of the chapter. For information on the state-of-the-art inspection technology, the reader is referred to the scientific and engineering literature or the Internet. A good place to start any search for the latest NDE technology is the home page of The American Society for Nondestructive Testing.[34]

2 LIQUID PENETRANTS

The liquid penetrants are used to detect surface connected discontinuities, such as cracks, porosity, and laps in solid, nonporous materials.[16] The method uses a brightly colored visible or fluorescent penetrating liquid, which is applied to the surface of a cleaned part. After a ‘‘dwell time’’ the liquid enters the discontinuity and then is removed from the part in a cleaning process. The penetrant is drawn from the flaw to the surface by a developer to provide an indication of surface-

Table 1 Capabilities of the Common NDI Methods

Method	Typical Flaws Detected	Typical Application	Advantages	Disadvantages
Radiography	Voids, porosity, inclusions, and cracks	Castings, forging, weldments, and structural assemblies	Detects internal flaws; useful on a wide variety of geometric shapes; portable; provides a permanent record	High cost; insensitive to thin laminar flaws, such as tight fatigue cracks and delaminations; potential health hazard
Liquid penetrants	Cracks, gouges, porosity, laps, and seams open to a surface	Castings, forging, weldments, and components subject to fatigue or stress–corrosion cracking	Inexpensive; easy to apply; portable; easily interpreted	Flaw must be open to an accessible surface, level of delectability operator dependent
Eddy current inspection	Cracks, and variations in alloy composition or heat treatment, wall thickness, dimensions	Tubing, local regions of sheet metal, alloy sorting, and coating thickness measurement	Moderate cost, readily automated; portable	Detects flaws that change conductivity of metals; shallow penetration; geometry sensitive
Magnetic particles	Cracks, laps, voids, porosity, and inclusions	Castings, forging, and extrusions	Simple; inexpensive; detects shallow subsurface flaws as well as surface flaws	Useful on ferromagnetic materials only; surface preparation required, irrelevant indications often occur; operator dependent
Thermal testing	Voids or disbonds in both metallic and nonmetallic materials, location of hot or cold spots in thermally active assemblies	Laminated structures, honeycomb, and electronic circuit boards	Produces a thermal image that is easily interpreted	Difficult to control surface emissivity and poor discrimination between flaw types.
Ultrasonic methods	Cracks, voids, porosity, inclusions and delaminations and lack of bonding between dissimilar materials	Composites, forgings, castings, and weldments and pipes	Excellent depth penetration; good sensitivity and resolution; can provide permanent record	Requires acoustic coupling to component; slow; interpretation of data is often difficult

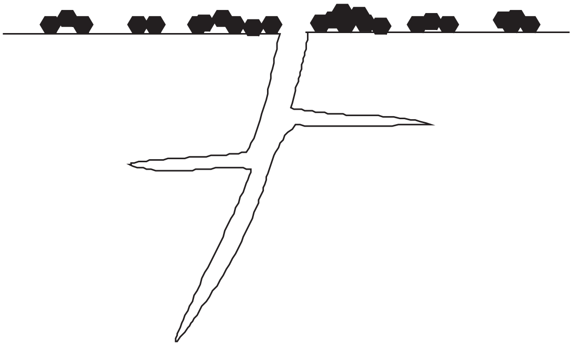

Fig. 1 Schematic representation of a part surface before cleaning for penetrant inspection.

connected defects. This process is depicted schematically in Figs. 1–4. A penetrant flaw indication in turbine blade is shown in Fig. 5.

2.1 Penetrant Process

Both technical societies and military specifications require a classification system for penetrants. Society documents (typically ASTM E165) categorize penetrants into visible and fluorescent, depending on the type of dye used. In each category there are three types, depending on how the excess penetrant is removed from the part. These are water washable, postemulsifiable, and solvent removable.

The first step in penetrant testing (PT) or inspection is to clean the part (Figs. 1 and 2). Sometimes this critical step is the most neglected phase of the procedure. Since PT only detects flaws that are open to the surface, the flaw and part surface must be free of dirt, grease, oil, water, chemicals, and other foreign materials that might block the penetrant's entrance to a defect. Typical cleaning procedures use vapor degreasers, ultrasonic cleaners, alkaline cleaners, or solvents.

After the surface is clean, a liquid penetrant is applied to the part by dipping, spraying, or brushing. In this step the penetrant on the surface is wicked into the flaw—see Fig 3. In the case of tight surface openings, such as fatigue cracks, the penetrant must be allowed to remain on the part for a minimum of 30 min

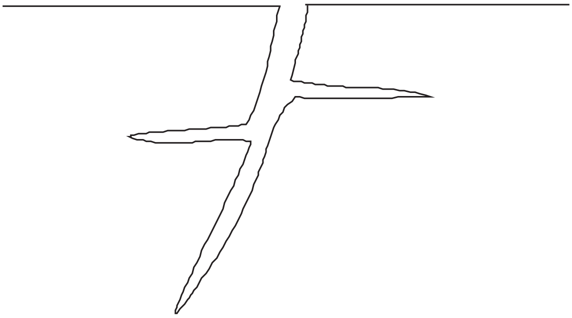

Fig. 2 Part surface after cleaning and before penetrant application.

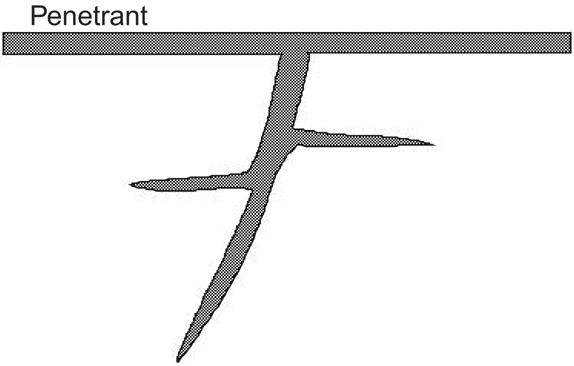

Fig. 3 Part after penetrant application.

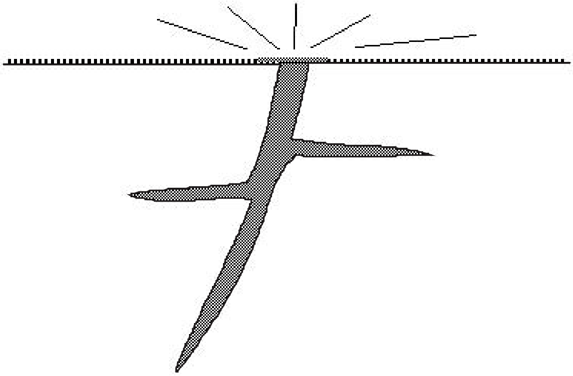

Fig. 4 Schematic representation of part after excess penetrant has been removed and developer applied.

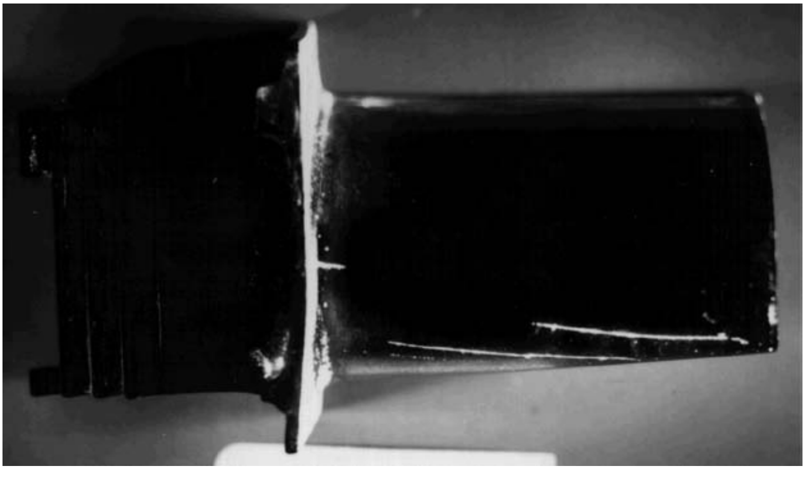

Fig. 5 Penetrant indication of a crack running along the edge of a jet engine turbine blade. Ultraviolet illumination causes the extracted penetrant to fluoresce.

to enhance the probability of complete flaw filling. High-sensitivity fluorescent dye penetrants are used for this type of inspection.

At the conclusion of the dwell time on the part, the excess penetrant is removed by one of three processes, depending on the type penetrant. Ideally, only the surface penetrant is removed with the penetrant in the flaw left undisturbed—see Fig. 4.

The final step in a basic penetrant inspection is the application of a fine powder developer. This may be applied either wet or dry. The developer aids in wicking the penetrant from the flaw and provides a suitable background for easy flaw detection. The part is then viewed under a suitable light source—either ultraviolet or visible. A typical fluorescent penetrant indication for a crack in a jet engine turbine blade is shown in Fig. 5.

2.2 Reference Standards

Several types of reference standards are used to check the effectiveness of liquid-penetrant systems. One of the oldest and most often used methods involves applying penetrant to hard chromium-plated brass panels. The panel is bent so as to place the chromium in tension, producing a series of cracks in the plating. These panels are available in sets containing fine, medium, and coarse cracks. The panels are used to classify penetrant materials by sensitivity and to detect degrading changes in the penetrant process.

2.3 Limitations of Penetrant Inspections

The major limitation of liquid-penetrant inspection is that it can only detect flaws that are open to the surface. Other inspection methods must be used to detect subsurface defects. A factor that may inhibit the effectiveness of liquid-penetrant inspection is surface roughness. Rough surfaces are likely to produce false indications by trapping penetrant. Penetrant testing is also not suited for the inspection of porous materials because the penetrant in the pores obscures any flaw indications. Other penetrant-like methods are available for porous components—see filtered particle inspection in *Nondestructive Testing Handbook,* Vol. 2.

3 ULTRASONIC METHODS

Ultrasonic methods utilize high-frequency sound waves to inspect the interior of parts. Sound waves are mechanical or elastic waves that propagate in fluid and solid media. Ultrasonic inspection is analogous to the use of sonar to detect schools of fish or map the ocean floor.[8] Both the government and industry have developed standards for ultrasonic inspections. These include but are not limited to the ASTM specifications 214-68, 428-71, and 494-75 and military specification MIL-1-8950H. Acoustic and ultrasonic testing can take many forms. Ultrasonic testing (UT) ranges from simple coin tapping to the transmission and reception of very high frequency waves into a part in order to analyze its internal structure.

UT instruments operating in the frequency range between 20 and 500 kHz are referred to as "sonic" instruments, while above 500 kHz operate in the domain of "ultrasonic" methods. To generate and receive ultrasonic waves, a piezoelectric transducer is usually used to convert electrical signals to sound wave signals and vice versa. The usual form of this transducer consists of a piezoelectric crystal mounted in a waterproof housing that facilitates its electrical connection to a pulsar (transmitter) and a receiver. In the transmit mode a high-voltage, short-duration electrical pulse is applied to the crystal, causing it to rapidly change shape and emit an acoustic or mechanical pulse. In the receive mode, any sound waves or echoes returning from along the path of the emitted acoustic pulse compress the piezoelectric crystal producing an electrical signal that is amplified and processed by the receiver.

3.1 Sound Waves

Ultrasonic waves have several characteristics such as wavelength λ, frequency f, velocity v, pressure P, and amplitude a. The following relationship between wavelength, frequency, and sound velocity is valid for all waves, including sound waves.

$$f\lambda = v$$

For example, the wavelength of longitudinal ultrasonic waves with a frequency 2 MHz propagating in steel is 3 mm and the wavelength of shear waves is 1.6 mm.

The formula that relates the sound pressure to the particle amplitude, with the terms as defined in the previous paragraph, is

$$P = 2\pi f \rho v a$$

Ultrasonic waves are reflected from boundaries or interfaces that separate a different materials or media. Each media has characteristic acoustic impedance, and reflections occur in a phenomenon quite similar to those observed with electrical signals. The acoustic impedance, Z, of any media capable of supporting sound waves is defined by

$$Z = \rho v$$

where ρ is the density of the media in g/cm^3, and v is the velocity of sound along the direction of propagation. Materials with high acoustic impedance are often referred to as sonically hard in contrast to sonically soft materials with low impedances. For example, steel ($Z = 7.7$ g/cm^3 $\times$ 5.9 km/s $= 45.4 \times 10^6$ kg/m^2 s) is sonically harder than aluminum ($Z = 2.7$ g/cm^3 $\times$ 6.3 km/s $= 17 \times 10^6$ kg/m^2 s). A list of acoustic properties of many common materials is provided in Appendix A.

3.2 Reflection and Transmission of Sound

Almost all the acoustic energy incident on air/solid interfaces is reflected because of the large impedance mismatch between air and most solids. For this reason, a coupling media, with impedance close to that of the part, is used to couple the sonic energy from the transducer into the part. A liquid couplant has obvious advantages for parts with a complex geometry, and water is the couplant of choice for most inspection situations. The receiver, in addition to amplifying the returning echoes, also gates the echoes that return between the front surface and rear surfaces of the component. Thus, any unusually occurring echo can either be displayed separately or used to set off an alarm.

A schematic diagram of a typical ultrasonic pulse echo setup is shown in Fig. 6. This method of displaying of voltage amplitude of the returning pulse versus time or depth (if acoustic velocity is known) at a single point of the specimen is known as an A-scan. In this figure, the first signal corresponds to a reflection from the front surface (FS) of the part and the last signal corresponds to the reflection from its back surface (BS). The signal or echo between FS and BS is from the defect in the middle of the part.

The portion of sound energy reflected from or transmitted through each interface is a function of the impedances of media on each side of that interface. The reflection coefficient *R* (ratio of the sound pressures or intensities of the reflected and incident waves) and transmission coefficient *T* (ratio of the sound pressures or intensities of the transmitted and incident waves) for an acoustic wave normally incident on to an interface are

$$R = \frac{p_t}{p_i} = \frac{Z_1 - Z_2}{Z_1 + Z_2}$$

$$R_{\text{pwr}} = \frac{I_r}{I_i} = \left(\frac{Z_1 - Z_2}{Z_1 + Z_2}\right)^2$$

Likewise, the transmission coefficients, T and T_{pwr}, are defined as

$$T = \frac{p_t}{p_i} = \frac{2 \times Z_2}{Z_1 + Z_2}$$

$$T_\rho = \frac{I_t}{I_i} = \frac{4 \times \left(\frac{Z_2}{Z_1}\right)}{\left[1 + \left(\frac{Z_2}{Z_1}\right)\right]^2}$$

where I_i, I_r, and I_t are the incident, reflected, and transmitted acoustic field intensities, respectively. Z_1 is the acoustic impedance of the media from which the sound wave is incident and Z_2 is impedance into which the wave is transmitted. From these equations one can calculate the reflection and transmission coefficients for a planar flaw containing air, $Z_1 = 450$ kg/cm^2 s, located in a steel part, $Z_2 = 45.4 \times 10^6$ kg/m^2 s. In this case, the reflection coefficient for the flaw is practically -1.0. The minus sign indicates a phase change of 180° for the reflected pulse (note that the defect signal in Fig. 6 is inverted or phase shifted by 180° from the front surface signal).

Effectively no acoustic energy is transmitted across an air gap, necessitating the use of water as a coupling media in ultrasonic testing. The acoustic properties of several common materials are shown in Appendix A. These data are useful for a number of simple, yet informative, calculations.

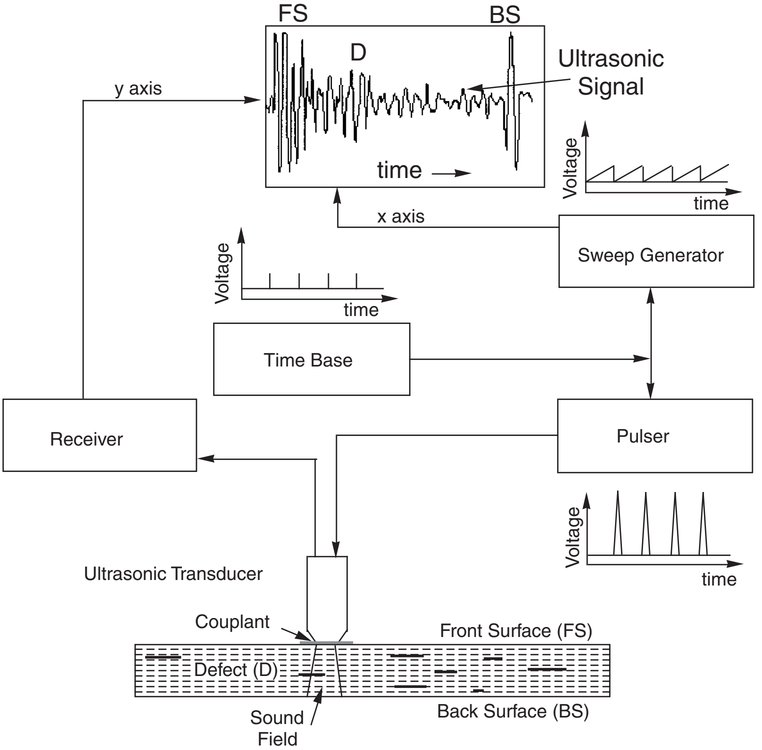

Fig. 6 A schematic representation of ultrasonic data collection and display in the A-scan mode.

Thus far our discussion has involved only longitudinal waves. This is the only wave type that travels through fluids such as air and water. The particle motion in this wave, if it were observed, is similar to the motion one would observe in a spring, or a Slinky toy, where the displacement and wave motion are collinear (the oscillations occur along the direction of propagation). The wave is referred to as compressional or dilatational since both compressional and dilatational forces are active in it. Audible sound waves are compressional waves. This wave type is observed in liquids and gases as well as in solids. However, a solid medium can also support additional types of waves such as shear and Rayleigh or surface waves. Shear or transverse waves have a particle motion that is analogous to what one sees in a rope that has been snapped. That is, the displacement of the rope is perpendicular to the direction of wave propagation. The velocity of this wave is about half that of compressional waves and is only found in solid media as indicated in the acoustic velocity appendix, i.e., Appendix A. Shear waves are often generated when a longitudinal wave is incident on a fluid/solid interface at angles of incidence other than 90°. Rayleigh or surface waves have elliptical wave motion, as shown in Fig. 7, and penetrate the surface for about one wavelength; therefore, they are used to detect surface and very near surface flaws. The velocity of Rayleigh waves is about 90% of the shear wave velocity. Their generation requires the special device shown in Fig. 8, which enables an

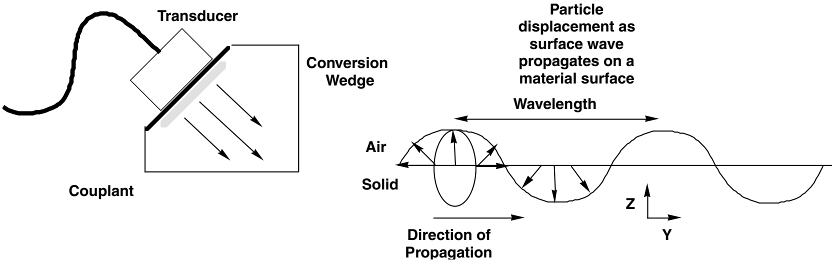

Fig. 7 Generation and propagation of surface waves in a material.

incident ultrasonic wave on the sample at a specific angle that is characteristic of the material (Rayleigh angle). The reader can find more details in Ref. 63.

3.3 Refraction of Sound

The direction of propagation of acoustic waves is described by the acoustic equivalent of Snell's law. Referring to Fig. 8, the directions of propagation are determined with the following equation:

$$\frac{\sin\,\theta_i}{c_\mathrm{I}} = \frac{\sin\,\theta_r}{c_\mathrm{I}} = \frac{\sin\,\gamma_r}{b_\mathrm{I}} = \frac{\sin\,\theta_t}{c_\mathrm{II}} = \frac{\sin\,\gamma_t}{b_\mathrm{II}}$$

where c_I is the velocity of the incident longitudinal wave, c_I and b_I are the velocities of the longitudinal and shear reflected waves, and c_II and b_II are the velocities of the longitudinal and shear transmitted waves in solid II. The angles of incidence and transmission are shown in Fig. 8. In the case of the water/steel

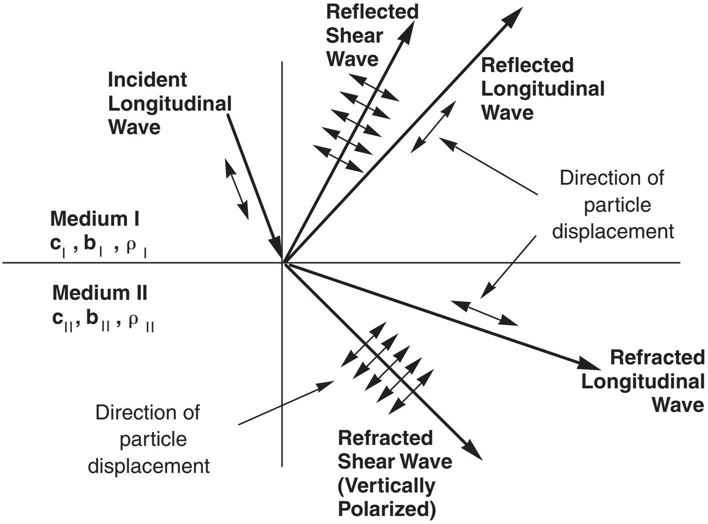

Fig. 8 Schematic representation of Snell's law and the mode conversion of a longitudinal wave incident on a solid/solid interface.

interface, there are no shear waves reflected because these waves do not propagate in fluids such as water. In this case, the above relationship is simplified. Since the water has a lower wave speed than either the compressional or shear wave speeds of the steel, the acoustic waves in the metal are refracted away from the normal. There will be a critical angle at which the refracted wave travels along the interface and does not enter the steel. Above this angle the acoustic wave will not be generated in the metal. A computer-generated curve is shown in Fig. 9 in which the normalized acoustic energy reflected and refracted at a water/steel interface are plotted as a function of angle of incidence. Note that the longitudinal or first critical angle for steel occurs at 14.5°. Likewise the shear or second critical angle occurs at 50°. If the angle of incidence is increased, above the first critical angle, only the shear wave is generated in steel and travels at an angle of refraction determined by Snell's law. Angles of incidence above the second critical angle produce a complete reflection of the incident acoustic wave; that is, no acoustic energy enters the solid. At a specific angle of incidence (Rayleigh angle) surface acoustic waves are generating on the material. The Rayleigh angle can be easily calculated from Snell's law by assuming that the refracted angle is 90°. The Rayleigh angle for steel is 29.5°. In the region between the two critical angles only the shear wave is generated and is referred to as "shear wave testing." There are two distinct advantages to inspecting parts with this type of shear wave. First, with only one type of wave present, the ambiguity that would exist concerning which type of wave is reflected from a defect does not occur. Second, the lower wave speed of the shear wave means that it is easier to resolve distances within the part. For these reasons, shear wave

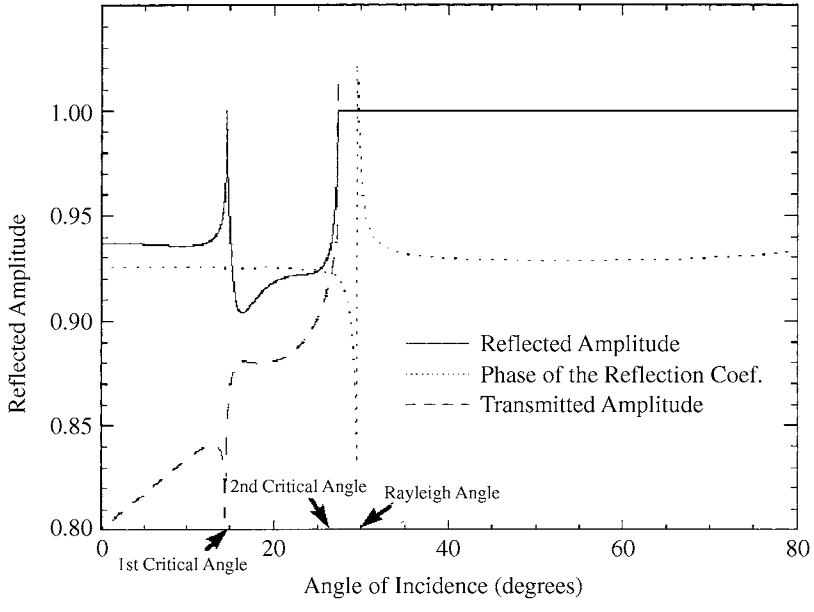

Fig. 9 The amplitude (energy flux) and phase of the reflected coefficient and transmitted amplitude versus angle of incidence for a longitudinal wave incident on a water–steel interface. The arrows indicate the critical angles for the interface.

inspection is often chosen for inspection of thin metallic structures such as those in aircraft.

Using the relationships for the reflection and transmission coefficients, a great deal of information can be deduced about any ultrasonic inspection situation with the acoustic wave incident at 90° to the surface. For other angles of incidence, computer software is often used to analyze the acoustic interactions. For more complicated materials or structures such as fiber-reinforced composites analytical predictions require the use of a rather complex analysis. In some cases, more complicated modes of wave propagation can occur. Examples of these include Lamb waves (plate waves), Stoneley waves (interface waves), Love waves (guided waves in the layer on a coated material), and others.

3.4 Inspection Process

Once the type of ultrasonic inspection has been chosen and the optimum experimental parameters determined, then one must choose the mode of presentation of the data. If the principle dimension of the flaw is less than the diameter of the transducer, then the A-scan method may be chosen, as shown in Fig. 6. The acquisition of a series of A-scans obtained by scanning the transducer in one direction across the specimen and displaying the data as distance versus depth is referred to as B-scan. This is the mode most often used by medial ultrasound instrumentation. In the A-scan mode, the size of the flaw may be inferred by comparing the amplitude of the defect signal to a set of standard calibration blocks. Each block has a flat bottom hole (FBH) drilled from one end. Calibration blocks have FBH diameters that vary in $\frac{1}{64}$-in. increments. Therefore, a number 5 block has a $\frac{5}{64}$-in. FBH. By comparing the amplitude of the signal from a calibration block with one from a defect, the inspector may specify a defect size as equivalent to a certain size FBH. The equivalent size is meaningful only for approximately smooth flaws that are nearly perpendicular to the path of the ultrasonic beam.

If the flaw size is larger than the transducer diameter, then the C-scan mode is usually selected. In this mode, shown in Fig. 10, the transducer is rastered back and forth in two directions across the part. In normal operation a line is traced on a monitor or piece of paper. When a flaw signal is detected between the front and back surface, a line with much increased darkness appears on the paper or monitor. Using this mode of presentation, a planar projection of each flaw is presented to the viewer and its positional relationship to other flaws and to the component boundaries is easily ascertained. Unfortunately, the C-scan mode does not show depth information, unless an electronic gate is set to capture only information from within a specified time window or time gate in the part. With current computer capability it is a rather simple matter to store all of the returning A-scan data and display only the data in a C-scan mode for specific depth. The C-scan mode provides a visual representation of a slice of the material at a certain depth and is very useful for inspection purposes.

Depending on the structural complexity and the attenuation of the signal by the material, flaws as small as 0.015 in. can be reliably detected and quantified using this inspection method. An example of a typical C-scan printout of an adhesively bonded test panel is shown in Fig. 11. While the panel was fabricated with a Teflon void-simulating implant in the center, the numerous additional white areas indicate the presence of a great deal of porosity in the part.[50,58]

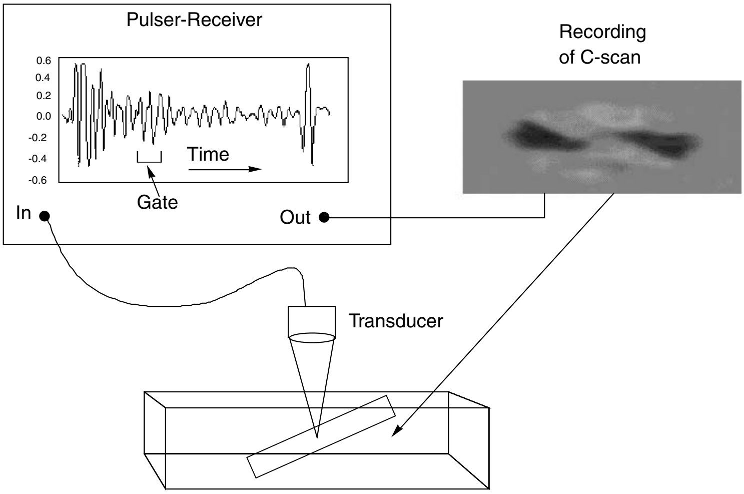

Fig. 10 Schematic representation of ultrasonic data collection. The data are displayed using the C-scan mode. The image shows a defect located at a certain depth in the material.

4 RADIOGRAPHY

In classical NDE radiography the projected X-ray attenuation for a multitude of paths through a specimen are recorded as a two-dimensional image on some type of recording media. In computed tomography (CT), the attenuation values are used to calculate a cross-sectional image of the component being inspected. A detailed description of radiography testing (RT) may be found in the *Nondestructive Testing Handbooks.*^{10}

The classic RT process is shown schematically in Fig. 12. Note that RT records visually any feature that changes the attenuation of the X-ray beam as it transverses a path through the component. This local change in attenuation produces a change in the intensity of the X-ray beam, which translates to a change in the density or darkness on a film or electronic recording device. The inspector uses this change in brightness to detect internal anomalies, which sometimes appear as a mere shadow on the radiograph. The inspector is greatly aided in detecting and quantifying flaws by knowing the geometry of the part and how this relates to the image. It should be noted in Fig. 12 that only flaws that change the attenuation of the X-ray beam on passage through the part are recorded. For example, a delamination in the laminated specimen is not visible because there is no change in attenuation of the X-ray beam. Flaws that are oriented parallel

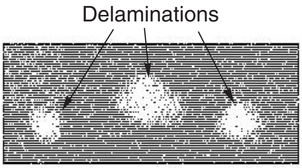

Fig. 11 Typical C-scan image of a composite specimen showing delaminations and porosity. Black and white are reversed.

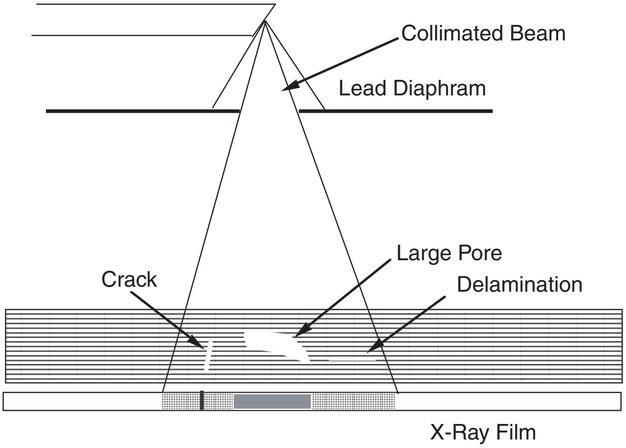

Fig. 12 Schematic radiograph of a typical composite with typical flaws.

to the X-ray path do not attenuate the X-ray beam and thus allow more radiation to expose the film and thus appear darker than the surrounding image. An example of a crack in the correct orientation to be visible on a radiograph of a piece of tubing is shown in Fig. 13.

4.1 Generation and Absorption of X-Radiation

X-radiation can be produced from a number of processes. The most common form of producing X-rays is with an electron tube in which a beam of energetic electrons impacts a metal target. As the electrons are rapidly decelerated in this collision, a wide band of X-radiation is produced, analogous to white light. This band of radiation is referred to as Bremsstrahlung or breaking radiation. Higher-energy electrons produce shorter-wavelength or more energetic X-rays. The relationship between the shortest-wavelength radiation and the highest voltage applied to the tube is given by

$$\lambda = \frac{12{,}336}{\text{voltage}}$$

where λ, in Ångstroms, is the shortest wavelength of the X-radiation produced.

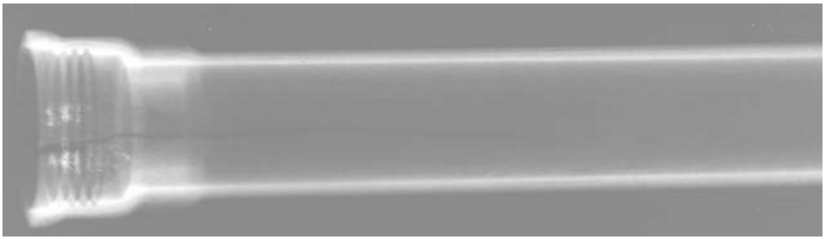

Fig. 13 Radiograph of a crack in the end of an aluminum tubing.

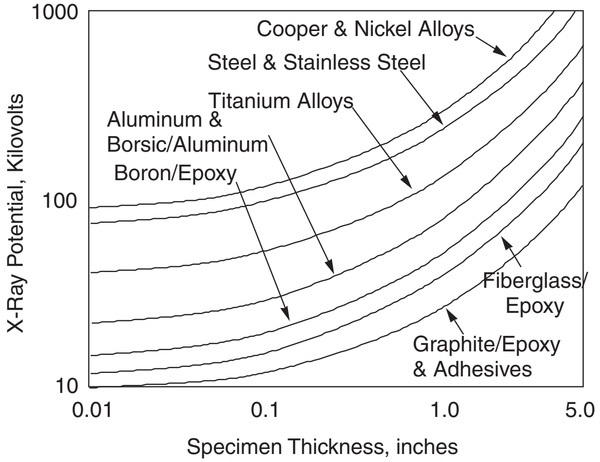

Fig. 14 Plot of the X-ray tube voltage versus thickness of several important industrial materials.

The more energetic the radiation, the more penetrating power it has. Therefore, higher energy radiation is used on dense materials such as metals. While it is possible to analytically predict what X-ray energy would provide the best image for a specific material and geometry, a simpler method of determining the optimum X-ray energy is shown by Fig. 14. Note that higher energies are used for dense materials, e.g., steels, or for thick light materials, e.g., large plastic parts. An alternative method to using this figure is to use radiographic equivalence factors shown in Table 2.[64] Aluminum is the standard material for voltages below 100 KeV, while steel is the standard above this voltage. When radiographing any other material, its thickness is multiplied by the factor to obtain the equivalent thickness of the standard material. The radiographic parameters are set for this thickness of aluminum or steel. In this manner a good-quality radiograph is

Table 2 Approximate Radiographic Equivalence Factors[64]

	100 kV	150 kV	220 kV	250 kV	400 kV	1 MeV	2 MeV	4–25 MeV	192Ir	60Co
Metal										
Magnesium	0.05	0.05	0.08							
Aluminum	0.08	0.12	0.18						0.35	0.35
Aluminum alloy	0.10	0.14	0.18						0.35	0.35
Titanium		0.54	0 54		0.71	0.9	0.9	0.9	0.9	0.9
Iron/all steels	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
Copper	1.5	1.6	1.4	1.4	1.4	1.1	1.1	1.2	1.1	1.1
Zinc		1.4	1.3		1.3			1.2	1.1	1.0
Brass		1.4	1.3		1.3	1.2	1.1	1.0	1.1	1.0
Inconel X		1.4	1.3		1.3	1.3	1.3	1.3	1.3	1 3
Monel	1.7		1.2							
Zirconium	2.4	2.3	2.0	1.7	1.5	1.0	1.0	1.0	1.2	1.0
Lead	14.0	14.0	12.0			5.0	2.5	2.7	4.0	2.3
Halfnium			14.0	12.0	9.0	3.0				
Uranium			20.0	16.0	12.0	4.0		3.9	12.6	3.4

obtained for the part in question. For example, assume that one must radiograph a 0.75-in.-thick piece of brass with a 400 KeV X-ray source; then the inspector should multiply 0.75 by the factor of 1.3 to obtain 0.98. This means that an acceptable radiograph of the brass plates would be obtained with the same exposure parameters as would be used for 0.98 in. (approximately 1 in.) of steel.

Radiation for RT can also be obtained from the decay of radioactive sources. In this case, the process is usually referred to as gamma radiography. These radiation sources have several different characteristics from X-ray tubes. First, the gamma radiation is very nearly monochromatic; that is, the spectrum of radiation contains only one or two dominant energies. Second, the energies of most sources is in the million-volt range, making this source ideal for inspecting highly attenuating materials and structures. Third, the small size of these sources permits them to be used in tight locations where an X-ray tube could not fit. Fourth, since the gamma-ray source is continually decaying, adjustments to the exposure time must be made to achieve consistent results over time. Finally, the operator must always remember that the source is continually on and is therefore a persistent safety hazard. Aside from these differences, gamma radiography differs little from standard practice, so no further distinction between the two will be given.

4.2 Neutron Radiography

Neutron radiography[65] may be useful in inspecting materials and structures in some circumstances. Because the attenuation of neutrons is not related to atomic number in a regular manner, some elements can be more easily detected that others. While X-rays are most heavily absorbed by high-atomic-number elements, this is not true of neutrons, as shown in Fig. 15. The reader can discern that hydrogen adsorbs neutrons more strongly than do most metals. This means that any hydrogen-containing materials would be easily detected within a heavy metal container. Neutron radiography has been used to detect porosity in the

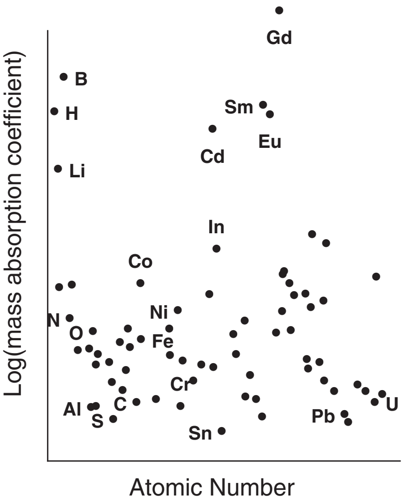

Fig. 15 Plot of mass absorption coefficient versus atomic number.

adhesive used to join metal structures. In this case, the hydrogen of the adhesive is preferentially absorbed compared to the metal—see the schematic representation of a neutron radiograph of an adhesively bonded specimen with various flaws shown in Fig. 16.

Neutron radiography, however, does have several constraints. First, neutrons do not expose radiographic film and therefore fluorescing must be used to produce an image with light. The image produced in this manner is not as sharp and well defined as that from X-rays. Second, there does not exist a high-flux, portable source of neutrons. This means that a nuclear reactor is most often used to supply the penetrating radiation. Given these restrictions, there are times when there is no other alternative to neutron radiography, and utility of the method outweighs its expense and difficulty.

4.3 Attenuation of X-Radiation

An appreciation of how radiographs are interpreted requires a fundamental understanding of the X-ray absorption. The basic relationship governing this phenomenon is de Beer's law.

$$I = I_0 e^{-\mu x}$$

where I and I_0 are the transmitted and incident X-ray beam intensities, respectively, μ is the attenuation coefficient of the material in reciprocal centimeters (cm^{-1}), and x is the thickness of the specimen, in centimeters. Since the attenuation coefficient is a function of both the composition of the specimen and the wavelength of the X-rays, it would be necessary to calculate or measure it for each wavelength used in radiographs testing (RT). However, it is possible to calculate the attenuation coefficient of a material for a specific X-ray energy using the mass absorption coefficient, μ, as defined below. The mass absorption coefficients for most elements are readily available for a variety of X-ray energies[10]

$$\mu_m = \frac{\mu}{\rho}$$

where μ is the attenuation coefficient of one element of the material, in reciprocal cm, and ρ its density in $\mathrm{g/cm^3}$. The mass absorption coefficient for the material is obtained, at a specific X-ray energy, by multiplying μ_m of each ele-

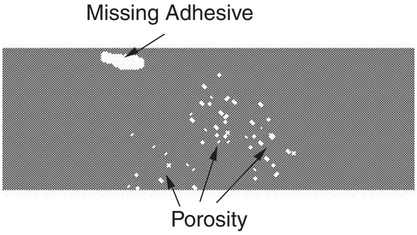

Fig. 16 Neutron radiograph of specimen showing bond line flaws.

ment by its weight fraction in the material and summing these quantities. Multiplying this quantity by the density of the material yields its attenuation coefficient. This procedure is not often used in practice because the results are valid only for a narrow band of wavelengths. In practice, radiographic equivalency factors are used instead. This process points out that each element in a material contributes to the attenuation coefficient by an amount proportional to its atomic percentage in the composition.

4.4 Film-Based Radiography

The classical method of recording an X-ray image is with film. Because of the continued importance of this medium of recording and the fact that much of the technology associated with it is applicable to newer solid-state recording methods, this next section explores film radiography in some detail.

The relationship between the darkness produced on an X-ray film and the quantity of radiation falling on it is shown in Fig. 17. This is a log-log plot of darkness or film density and relative exposure. Varying the time of exposure, intensity of the beam, or specimen thickness will change the film density. The slope of the curve along its linear portion is referred to as the film gamma, γ. Film has characteristics that are quite analogous to electronic devices. For example, the greater the gamma or amplification capability of the film, the smaller its dynamic range—the range of exposures over which density is linearly related to thickness. If it is necessary to use a high gamma film to detect very subtle flaws in a part with a wide range of thickness, then it will be necessary to expose several different film types in the same cassette—see Fig. 18. In this way, each film will be optimized for flaw detection in a narrow range of thickness of the part.

Using what we have learned so far, it is possible to calculate the minimum detectable flaw size for a specific RT situation. A method is even available to check the radiographic procedure to determine if it has achieved the calculated capability. This procedure does not ensure that the radiograph was taken with the specimen in the proper orientation; it only provides a method of checking the radiographic procedures for proper execution. This is covered in the section on penetrameters (Section 4.5).

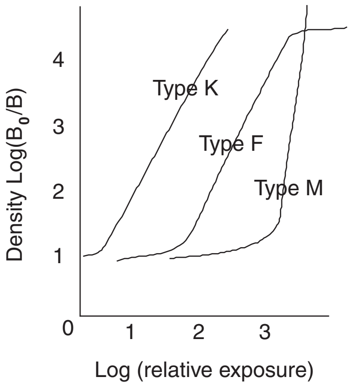

Fig. 17 Density or darkness of X-ray film versus relative exposure for three common films.

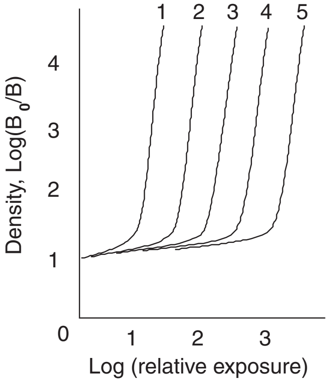

Fig. 18 Density versus relative exposure for films that could be used in a multiple film exposure to obtain optimum flaw delectability in a complex part.

Using knowledge of the minimum density difference that is detectable by the average radiographic inspector, the following relationship may be derived that relates the radiographic sensitivity S to both RT parameters and thickness changes in the specimen:

$$S = \frac{2.3}{\gamma \times \mu \times x}$$

where S is the radiographic sensitivity in percent, γ is the film gamma for the exposure conditions used, μ is the attenuation coefficient of the specimen material, and x is the maximum thickness of the part associated with the radiographic film. To check the RT process to determine if this sensitivity was achieved the radiographer uses a penetrameter.

4.5 The Penetrameter

An example of a penetrameter is shown schematically in Fig. 19, while its image on a radiograph is shown schematically in Fig. 20. This type of penetrameter was chosen because it is easily related to the previous discussion of radiographic sensitivity. This penetrameter is quite simply a thin strip of metal or polymeric material in which three holes of varying sizes are machined or punched. It is composed of the same material as the specimen and has a thickness of 1, 2, or 4% of its maximum thickness. The holes in the penetrameter have diameters that are 1, 2, and 4 times its thickness. The sensitivity achieved for each radiographic is determined by noting the smallest hole just visible in the thinnest

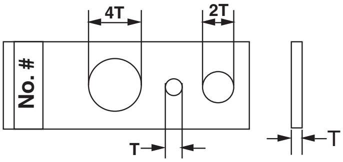

Fig. 19 Schematic of typical film penetrameter.

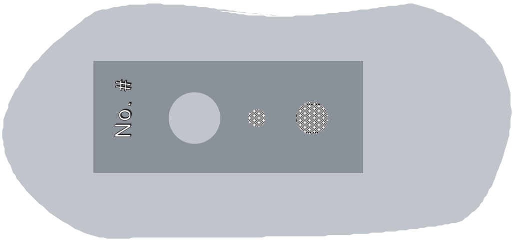

Fig. 20 Radiograph of the penetrameter shown in Fig. 19. The 1T hole is just visible indicating the resolution obtained in the radiograph.

penetrameter on a film and by using Table 3. By calculating the radiographic sensitivity and then noting the level achieved in practice, the radiographic process can be quantitatively evaluated. While this procedure does not offer any guarantee of flaw detection, it is quite useful in evaluating the effectiveness the RT process.

Almost all variables of the radiographic process may be easily and rapidly changed with the aid of tables, graphs, and nomograms, which are usually provided by film manufacturers free of charge. For more information in this regard the reader is referred to the commercial literature.

4.6 Real-Time Radiography

While film radiography represents the bulk of the radiographic NDE performed at this time, new methods of both recording the data and analyzing it are coming into widespread usage. For example, filmless radiography (FR) or real-time (RT) radiography testing uses solid-state detectors or television detection and image-processing methods instead of film to record the image. These methods have several advantages along with some disadvantages. For example, FR permits viewing a radiographic image while the specimen is being moved. This often permits the detection of flaws that would normally be missed in conventional film radiography because of the limited number of views or exposures usually taken—remember that the X-ray beam must pass along a cracklike flaw in order to be detectable. Additionally, the motion of some flaws enhances their detectability because they present the inspector with a different image as a function of time than he or she is accustomed to viewing. Additionally, image enhance-

Table 3 Sensitivity versus the Smallest Hole Just Visible in the Thinnest Penetrameter

Sensitivity, *S* (%)	Quality Level (%T–Hole Diameter)
0.7	1–1T
1.0	1–2T
1.4	2–1T
2.0	2–2T
2.8	2–4T
4.0	4–2T

ment techniques can now be economically applied to these images because of the availability of inexpensive, fast computing hardware. The price to be paid for these advantages is the lower resolution of the FR image compared to film. Typical resolution capabilities of FR systems are in the range from 4 to perhaps 20 line pairs/mm, while film resolution capabilities are in the range from 10 to 100 line pairs/mm. This means some very fine flaws may not be detectable with FR and the inspector must resort to film. However, in cases where resolution is not the limiting factor in delectability, the benefits of software-enabled image enhancement can be significant. While the images on film may also be enhanced using the image-processing schemes, they cannot be performed in real or near real time, as can be done with a totally electronic system.

4.7 Computed Tomography

Another advance in industrial radiography with and unrealized capability to link NDE with engineering design and analysis is computed tomography (CT). The principle advantage of this method is that it produces an image of a thin slice of the specimen under examination. This slice is parallel to the path of the X-ray beam as it passes through the specimen as contrasted to the image produced by classical radiography, where the image is formed on a plane perpendicular to the path of the X-ray beam on passage through the specimen. The classical radiographic image can be difficult to interpret because it collapses all of the image information in the specimen between the source of X-rays and the recording media, whereas the CT image is computed or calculated from X-ray intensity data and does not contain information from planes outside the thin slice.

This comparison is best explained with images with these two modalities. Figure 13 shows a typical radiograph where one can easily see the image of the top and bottom surfaces of the tube under inspection. Contrast this with the image in Fig. 21 that shows a CT slice through a flashlight. The individual

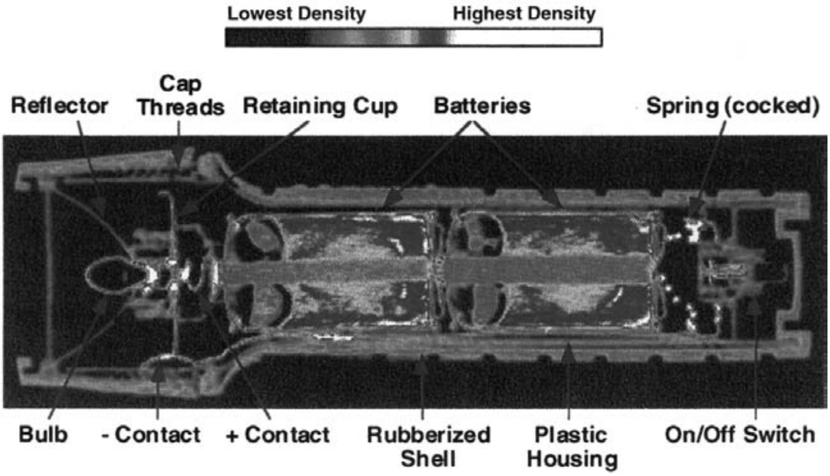

Fig. 21 Computed tomography of flashlight.

components of the flashlight are easily visible and any misplacement of its components or defects in its assembly could be easily detected. An image on a finer scale than this can clearly reveal microstructural details of a material as shown in Fig. 22. Everyone will recognize that this is a pencil. Not only are the key features clearly visible, but even the growth rings of the wood are clearly visible. In fact, the details of the growth during each season are visible as rings within rings. The information in the CT image contrasted with conventional radiographs is striking. First, the delectability of a defect is independent of its position in the image. This is not the case with the classical radiograph where the defect delectability decreases significantly with depth in the specimen because the defect represents a smaller change in the attenuation of the X-ray beam as the depth increases. Second, the defect delectability is very nearly independent of its orientation. This again is clearly not the case with classical radiography. The extent to which CT could alter radiography is only now being explored. New applications are being discovered frequently. For example, with the computed digital image of CT it is possible to search for various flaw conditions using computer analysis and relieve the inspector of much of the tedium of examining structures for the odd flaw. In addition, it is possible to link the digital CT image with finite-element analysis software to examine precisely how the flaws present will affect such parameters as the stress distribution, heat flow, etc. With little effort one could analyze the full three-dimensional performance of many engineering structures.

5 EDDY CURRENT INSPECTION

Eddy current methods are used principally to inspect electrically conducting components for near-surface flaws such as cracks and voids, heat treatment condition, external dimensions and wall thickness of thin-walled tubing, and thickness of nonconducting coatings on a metallic substrate.[17] Quite often several of these conditions can be monitored simultaneously if instrumentation capable of multiple frequencies is used and the results are analyzed using computer algorithms.

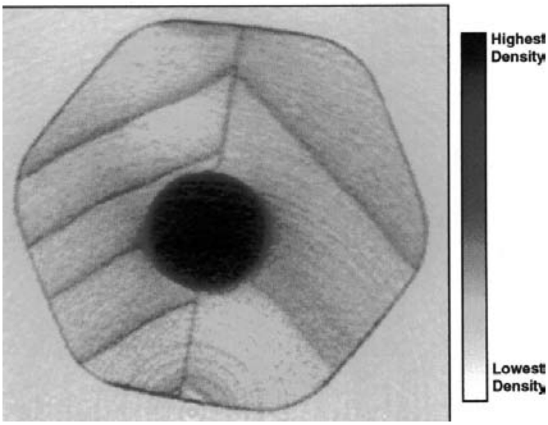

Fig. 22 Computed tomograph of a pencil. The reader will note the yearly growth rings and even the growth variations within a single growing season.

This NDE method is based on the principle that eddy currents are induced in a conducting material when a coil or array of conductors (probe) with an alternating or pulsating electric current is placed in close proximity to the surface of the material. The induced currents create an electromagnetic field that opposes the field of the inducing coil in accordance with Lenz's law. The eddy currents circulate in the part in closed, continuous paths, and their magnitude depends on several variables. The first is the magnitude and frequency of the current in the inducing coil. Second, is the coil's shape and position relative to the surface of the part, the electrical conductivity, magnetic permeability, and shape of the part. Finally, there are the influences of any discontinuities or inhomogenities within the material. Therefore, the eddy currents are useful in exploring these properties of test materials, as well as any discontinuities or variations in geometry and dimensions of materials.

5.1 Skin Effect

Since alternating currents are necessary to eddy current inspection, information from the inspection is limited to the near-surface region by the skin effect. Within the material, the eddy current density decreases rapidly with distance from the surface. The density of the eddy current field falls off exponentially with depth and diminishes to a value of about 37% of the at-surface value at a depth referred to as the standard depth of penetration (SDP). The SDP in meters may be calculated with the simple formula

$$\mathrm{SDP} = \frac{1}{\sqrt{\pi f \sigma \mu}}$$

where f is the test frequency in hertz, σ is the test material's electrical conductivity in mho/m (see Appendix B for a table of conductivities for common metals), and μ is its permeability in H/m. This latter quantity is the product of the relative permeability of the specimen, 1.0 for nonmagnetic materials, and the permeability of free space, which is $4\pi \times 10^{-7}$ H/m.

5.2 Impedance Plane

While the SDP is used to give an indication of the depth from which useful information can be obtained, the choice of the independent variables in most test situations is usually made using the impedance plane diagram suggested by Forster.[66] It is theoretically possible to calculate the optimum inspection parameters from numerical codes based on Maxwell's equations, but this is a tedious task that is justified only in special situations.

The eddy currents induced at the surface of a material are time varying and have amplitude and phase. The complex impedance of the coil used in the inspection of a specimen is a function of a number of variables. The effect of changes in these variables can be conveniently displayed with the impedance diagram that displays the variations in amplitude and phase of the coil impedance as functions of the dependent variables specimen conductivity, thickness, and distance between the coil and specimen or lift-off. For the case of an encircling coil on a solid cylinder, shown schematically in Fig. 23, the complex impedance plane is displayed in Fig. 24. The reader will note that the ordinate and abscissa

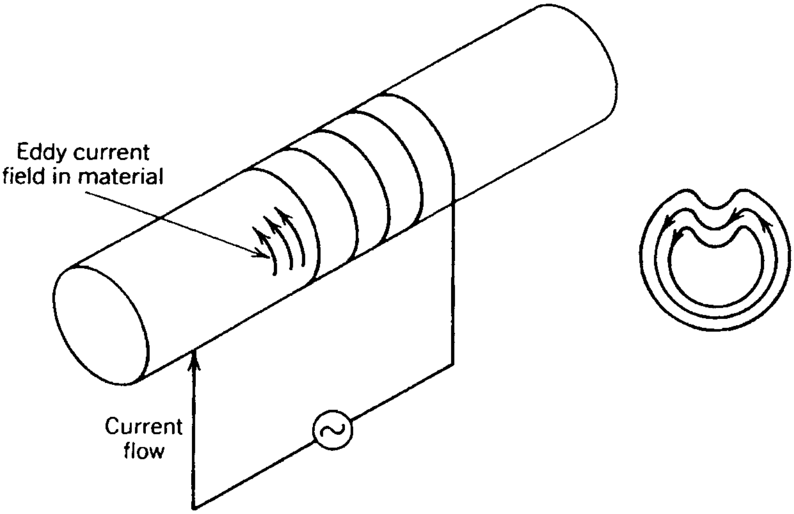

Fig. 23 Schematic representation for an eddy current inspection of a solid cylinder. Also shown are the eddy current paths within the cross section of the cylinder in the vicinity of the crack.

are normalized by the inductive reactance of the empty coil. This eliminates the effects of the coil/specimen geometry. The numerical values are shown on the large curve, called reference numbers. These numbers are used to combine the effects of conductivity and size of the test specimen and the frequency of the measurement into a single parameter. In this way, the diagram is useful for most test conditions encountered by the NDE inspector. The reference numbers shown on the outermost curve are obtained with the following relationship, for nonmagnetic materials:

$$\text{Reference \#} = r\sqrt{2\pi f \mu \sigma}$$

where r is the radius of the bar in meters, f is the frequency of the test in hertz, μ is the magnetic permeability of free space ($4\pi \times 10^{-7}$ H/m), and σ is the conductivity of the specimen in mho/m. The outer curve in Fig. 24 is useful only for the case where the coil is the same size as the solid cylinder, which almost never happens. For those cases when the coil is larger than the test specimen, which is usually the case, a coil-filling factor is calculated. This is quite easily accomplished with the following formula:

$$N = \left(\frac{\text{diameter}_{\text{specimen}}}{\text{diameter}_{\text{coil}}}\right)^2$$

Figure 24 shows the impedance plane with a curve for a cylinder/coil inspection geometry with a fill factor of 0.75. Note that the reference numbers on the ordinate representing the different fill factors can be determined by projecting a

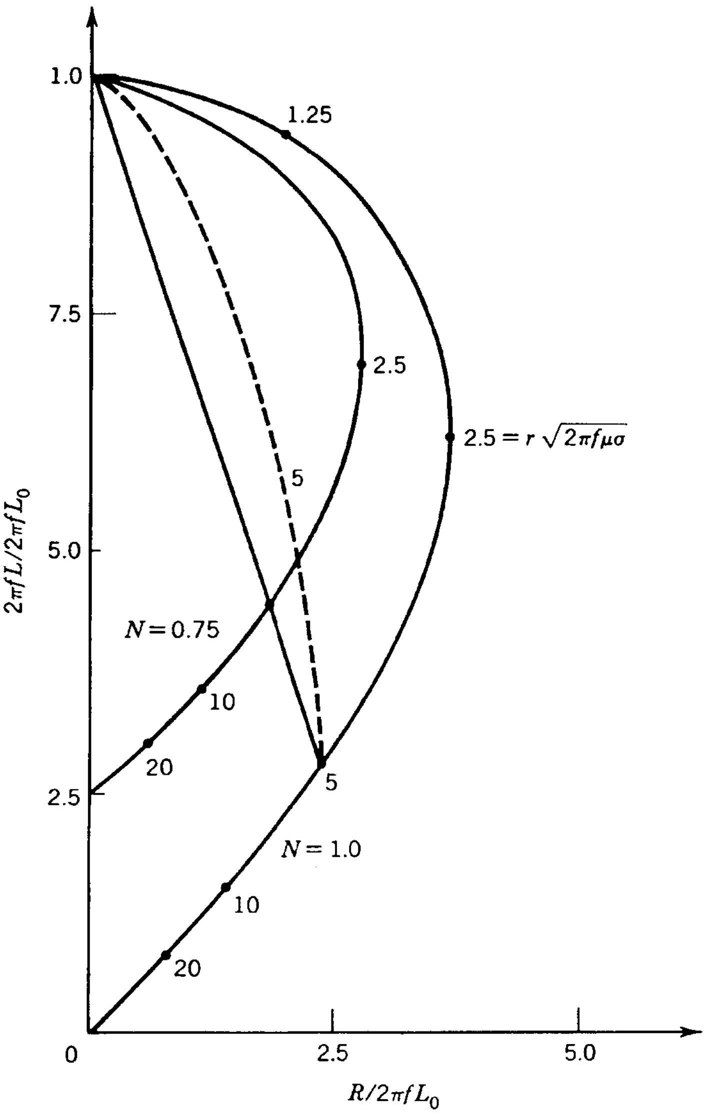

Fig. 24 Normalized impedance diagram for a long encircling coil on a solid, nonferromagnetic cylinder. For $N = 1$ the coil and cylinder have the same diameter, while for $N = 0.75$ the coil is approximately 1.155 times larger than the cylinder.

straight line from point 1.0 on the ordinate to the reference number of interest. This is shown for reference number 5.0. Both the fill factor and the reference number change when the size of either the specimen or coil changes. This is shown in Fig. 24 for solid cylinders. The other geometry of general interest is the thin-walled tube—remember that the skin effect limits the thickness of a conductor that may be inspected.

For an infinitely thin-walled tube the impedance plane is shown in Fig. 25. Also included in this figure is the curve for a solid cylinder. The curve segments

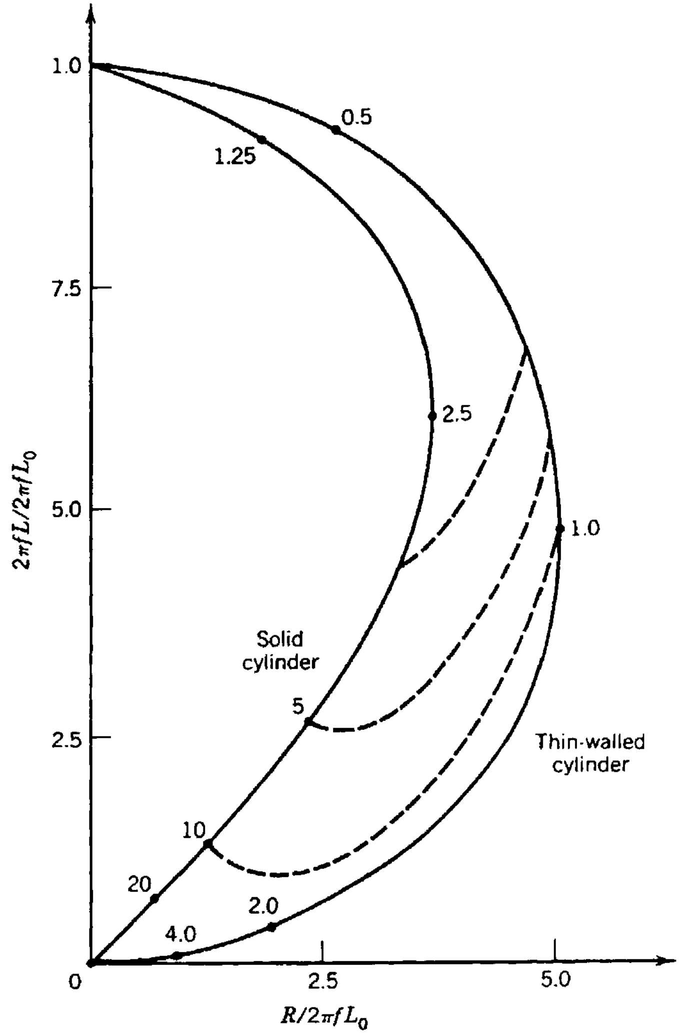

Fig. 25 Normalized impedance diagram for a long encircling coil on both a solid and thin-walled conductive but nonferromagnetic cylinders. The dashed lines represent the effects of varying wall thickness.

that connect these two extreme cases are for walled cylinders of varying thickness. The semicircular curve for the thin cylinder is used in the same manner as described above.

5.3 Lift-Off of Inspection Coil from Specimen

In many inspection situations the only independent variables are frequency and lift-off. High frequencies are used for detecting defects such as surface cracks or corrosion, and low frequencies are mostly used to detect deeply buried defects. While it is possible to change the coil shape and measurement configuration, the reader is referred to the literature for a discussion of the effects of these more complex variables. The relationships discussed thus far would seem of little practical usefulness. However, if a small section of Fig. 25 is expanded as shown in Fig. 26, then their utility of a well-chosen frequency will become apparent. In this figure, vectors represent small changes in thickness, lift-off, and conductivity. Since these vectors all point in different directions, the signals that they represent have different phases. Thus, instrumentation with phase discrimination circuitry can differentiate among these different conditions. Changes in conductivity can arise from several different conditions, the most important of which is due to material inhomogeneity. For example, aluminum alloys can have quite different conductivities depending either on their heat treatment or the presence of cracks or voids. These flaws decrease the apparent conductivity of the specimen because the eddy currents must either travel a longer distance to complete their circuit or the material presents a more resistive path. There are other important eddy current inspection capabilities that cannot be covered in this brief description of this important tool. These include the inspection of ferromagnetic alloys, plate and sheet stock, and the measurement of film thickness on metallic substrates. For a treatment of these and other special applications of eddy current NDE, the reader is referred to the *Nondestructive Testing Handbook,* Vol. 4, *Electromagnetic Testing.*[17]

There are many methods of making and displaying eddy current measurements. Two of the more common methods are shown schematically in Fig. 27. In the absolute coil arrangement very accurate measurements can be made of the difference between the two different samples. One can be a standard, while the other is the component in question. In the differential coil arrangement the differences between two variables at two slightly different locations are compared. For this arrangement slightly varying dimensions and conductivity are not sensed, while singularities such as cracks or voids are highlighted even in the presence of other more slowly changing variables. The electronic circuitry used to accomplish each of these measurements can vary dramatically depending on the inspection. This aspect of eddy current testing will not be covered here, and the reader is referred to the manufacturers and engineering literature for details of the appropriate electronic circuitry.

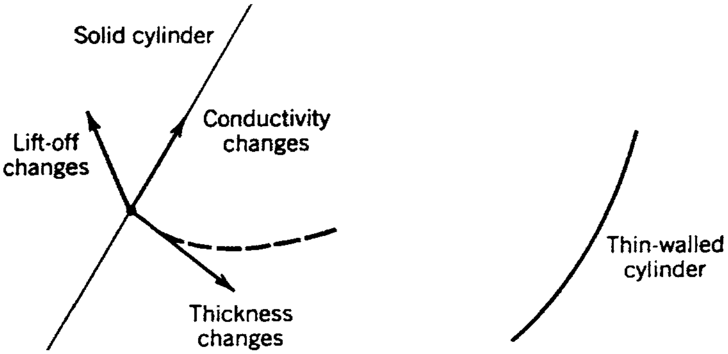

Fig. 26 Effects of various changes in inspection conditions on local signal changes in the impedance plane of Fig. 25. Phase differentiation is relatively easily accomplished with current instrumentation.

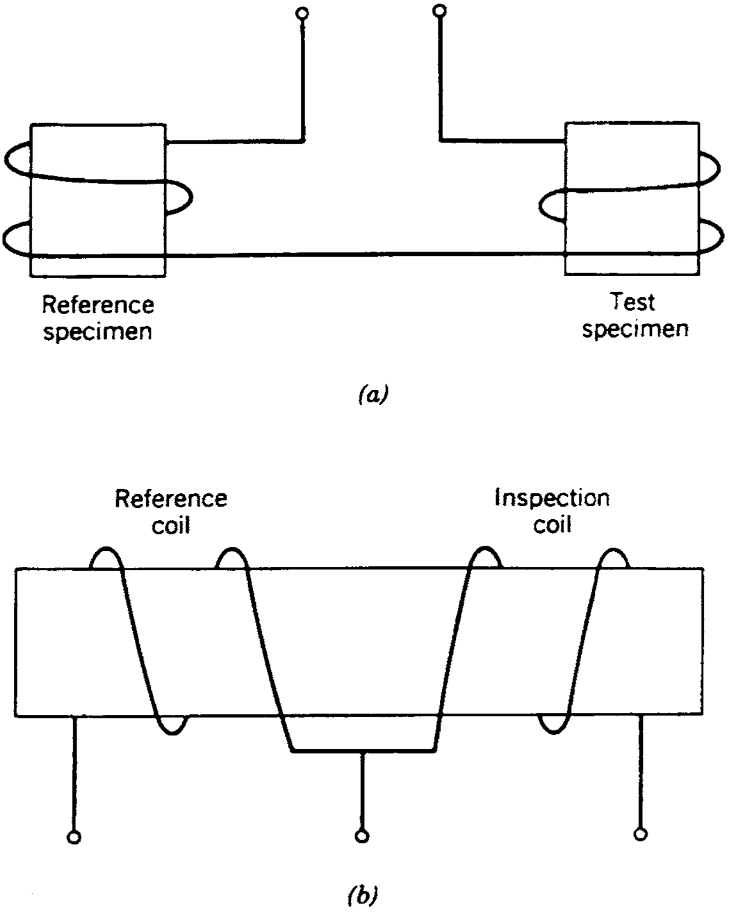

Fig. 27 Schematic representation of an (*a*) absolute versus (*b*) a differential coil configurations used in eddy current testing.

6 THERMAL METHODS

Thermal nondestructive inspection methods involve the detection of infrared energy emitted from the surface of a test object.[14] This technique is used to detect the flow of thermal energy either into or out of a specimen and the effect of anomalies in the surface temperature distribution. Other material properties that can influence this method are heat capacity, density, thermal conductivity, and emissivity of the test specimen. Defects that are usually detected include porosity, cracks, and delaminations. The sensitivity of any thermal method is greatest for near-surface flaws and degrades rapidly for flaws buried in thermally conductive materials. Materials with lower thermal conductivity yield better resolution because they produce larger thermal gradients.

6.1 Infrared Cameras

All objects emit infrared (IR) radiation with a temperature above absolute zero. At room temperature, the thermal radiation is predominately infrared, and measurements are concentrated in the region near 10 μm. IR cameras are available that view large areas by scanning over a single, liquid-nitrogen-cooled detector using rotating mirrors. Since the IR images can be stored in digital form, image processing is easily performed and thermal images can be stored on magnetic or optical media. For many applications, a thermal image that shows the relative temperature across the surface of a specimen is sufficient to detect flaws. However, if absolute temperatures are required, the IR instrumentation must be calibrated to account for the surface emissivity of the test subject.

Thermography's ability to detect flaws is often affected by the type of flaw and its orientation with respect to the surface of the object. To have a maximum effect on the surface temperatures, the flaw must interrupt heat flow to the surface. Since a flaw can occur at any angle to the surface, the important parameter is its projected area to the field of view of the camera. Subsurface flaws such as cracks parallel to the surface of the object, porosity, and debonding of a surface layer are easily detected. Cracks that are perpendicular to the object surface can be very difficult to find using thermography.

Most thermal NDE methods do not have excellent spatial resolution due to spreading of thermal energy as it diffuses to the surface. The greatest advantage of thermography is that it can be a noncontact, remote-viewing technique requiring only line-of-sight access to one side of a test specimen. Large areas can be viewed rapidly, since scan rates for IR cameras run between 16 and 30 frames per second. Temperature differences of 0.02°C or less can be detected in a controlled environment.

6.2 Thermal Paints

A number of contact thermal methods are available to give the temperature distribution for surfaces. These methods involve applying a coating to the sample and observing a color change as the specimen is thermally cycled. Several different types of coatings are available that cover a wide temperature range. Temperature-sensitive pigments in the form of paints have been made to cover a temperature range from 40 to 1600°C. Thermal phosphors are organic compounds that emit visible light when exposed to ultraviolet (UV) radiation. (The amount of visible light is inversely proportional to temperature.) Thermochromic compounds and cholesteric liquid crystals change color over large temperature ranges. The advantages of these approaches are the simplicity of the test and the relatively low cost if only small areas are scanned.

6.3 Thermal Testing

Excellent results may be achieved if thermography is performed in a dynamic environment where the transient effects of a heat flow in test object can be monitored. This enhances detection of areas where different heat transfer rates occur. Applications involving steady-state conditions are more limited. Thermography has been successfully used in several different area of testing. In medicine it is used to detect tumors. In aircraft manufacture or maintenance it is used to detect debonding in layered structures. In the electronics industry it is used to detect poor thermal performance of circuit board components. Recently, it has been sometimes used to detect stress-induced thermal gradients around defects in dynamically loaded test samples. For more information on thermal NDE methods the reader is referred to *Nondestructive Testing Handbook,*[14] *Special Nondestructive Testing Methods,*[21] and to *Metals Handbook: Nondestructive Evaluation and Quality Control.*[2]

7 MAGNETIC PARTICLE METHOD

The magnetic particle method of nondestructive testing is used to locate surface and subsurface discontinuities in ferromagnetic materials.[19] An excellent short reference for this NDE method is Ref. 9, especially Chapters 10–16. Magnetic particle inspection is based on the principle that magnetic lines of force, when present in a ferromagnetic material, are distorted by changes in material continuity, such as cracks or inclusions, as shown schematically in Fig. 28. If the flaw is open or close to the surface, the flux lines escape the surface at the site of the discontinuity. Even near-surface flaws, such as nonmagnetic inclusions, cause the same bulging of the lines of force above the surface. This distorted field, usually referred to as a leakage field, is used to reveal the presence of the discontinuity when fine magnetic particles are attracted to it. If these particles are fluorescent, their presence at a flaw will be visible under ultraviolet light much like penetrant indications—see Fig. 28. Magnetic particle inspection is used for the inspection of steel components because it is fast, easily implemented, and has rather simple flaw indications. The part is usually magnetized with an electric current, and then a solution containing fluorescent particles is applied by flowing it over the part. The particles that stick to the part form the indication of the flaw.

7.1 Magnetizing Field

The magnetizing field may be applied a component in any one of a number of methods. Its function is to generate a residual magnetic field in the surface of the part. The application of a magnetizing force (H) generates a magnetic flux (B) in the component as shown schematically in Fig. 29. In this figure, the magnetic flux density, B, has units of newtons/ampere or webers/m^2, and the strength of the magnetic field or magnetic flux intensity, H, has units of Oersted or amperes/meter. Starting at the origin, a magnetizing force is applied and the magnetic field internal to the part increases in a nonlinear fashion along the path shown by the arrows. If the force is reversed, then the magnetic field does not return to zero, but follows the arrows around the curve as shown. The reader will note that once the magnetizing force is removed, the flux density does not return to zero, but remains at an elevated value called the material's remanence. This is the point at which most magnetic particle inspections are performed. The reader will also note that an appreciable reverse magnetic force H must be

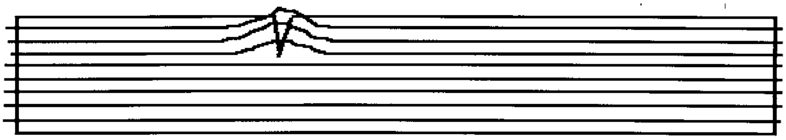

Fig. 28 Schematic representation of the magnetic lines of flux in a ferromagnetic metal near a flaw. Small magnetic particles are attracted to the leakage field associated with the flaw.

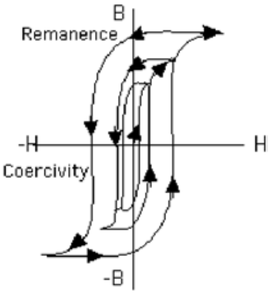

Fig. 29 Magnetic flux intensity, *H*, versus magnetic flux density, *B*, hysteresis curve for a typical steel. Initial magnetization starts at the origin and progresses as shown by the arrows. Demagnetization follows the arrows of the smaller hysteresis loops.

applied before the internal field density is again zero. This point $-H_c$ is referred to as the coercivity of the material. If the magnetizing force is applied and reversed, the material will respond by continually moving around this hysteresis loop.

Selection of the type of magnetizing current depends primarily on whether the defects are either open to the surface or are wholly below it. Alternating current (ac) magnetization is best for the detection of surface discontinuities because the current is concentrated in the near-surface region of the part. Direct currents (dc) magnetization is best suited for subsurface discontinuities because of the deeper penetration of the part. While dc can be obtained from batteries or dc generators, it is usually produced by half-wave or full-wave rectification of commercial power. Rectified current is classified as half-wave direct current (HWDC) or full-wave direct current (FWDC). Alternating current fields are usually obtained from conventional power mains, but it is supplied to the part at reduced voltage for reasons of safety and the high-current requirements of the magnetizing process.

Two general types of magnetic particles are available to highlight flaws. One type is a low-carbon steel with high permeability and low retentivity particle, which is used dry and consists of different sizes and shapes to respond to both weak and strong leakage fields. The other type of inspection media is very fine particles of magnetic iron oxide that are suspended in a liquid (either a petroleum distillate or water). These particles are smaller and have a lower permeability than the dry particles. Their small mass permits them to be held by the weak leakage fields at very fine surface cracks. Magnetic particles are available in several colors to increase their contrast against different surfaces or backgrounds. Dry powders are typically gray, red, yellow, and black, while wet particles are usually red, black, or fluorescent.

7.2 Continuous versus Noncontinuous Fields

Because the field is always stronger while the magnetizing current is on, the continuous magnetizing method is generally preferred. Additionally for specimens with low retentivity the continuous method is often used. In the continuous method, the current can be applied in short pulses, typically 0.5 s. The magnetic particles are applied to the surface during this interval and are free to move to the site of the leakage fields. Using liquid suspended fluorescent particles produces the most sensitive indications. For field inspections, the magnetizing current is often continuously applied during the test to give time for the powder to migrate to the defect site.

In the residual method, the particles are applied after the magnetizing current is removed. This method is particularly suited for production inspection of multiple parts.

The choice of direction of the magnetizing field within the part involves the nature of the flaw and its direction with respect to the surface and the major axis of the part. In circular magnetization, the field runs circumferentially around the part. It is induced into the part by passing current through it between two contacting electrodes. Since flaws perpendicular to the magnetizing lines are readily detectable, circular magnetization is used to detect flaws that are parallel or less than 45° to the surface of the long, circular specimens. Placing the specimen inside a coil to create a field running lengthwise through the part produces longitudinal magnetization. This induction method is used to detect transverse discontinuities to the axis of the part.

7.3 Inspection Process

The surface of the part to be examined should be clean, dry, and free of contaminants such as oil, grease, loose rust, loose sand, loose scale, lint, thick paint, welding flux, and weld splatter. Cleaning of the specimen may be accomplished with detergents, organic solvents, or mechanical means, such as, scrubbing or grit blasting.

Portable and stationary equipment are available for this inspection process. Selection of the specific type of equipment depends on the nature and location of testing. Portable equipment is available in lightweight units (35–90 lb), which can be readily taken to the inspection site. Generally, these units operate off of 115, 230, or 460 V ac and supply current outputs of 750–1500 A in half-wave or ac.

7.4 Demagnetizing the Part

Once the inspection process is complete, the part must be demagnetized. This is done by one of several ways depending on the subsequent usage of the component. A simple method of demagnetizing to remove any residual magnetism from small tools is performed as follows: Place the tool in the coil of a soldering iron and slowly withdrawn it. This has the effect of retracing the hysteresis loop a large number of times, each time with a smaller magnetizing force. When completely withdrawn, the tool will then have a very small remnant magnetic field, which is for all practical purposes zero. This same process is accomplished with industrial part by slowly reducing and reversing the magnetizing current till it is essentially zero. This process of reducing the residual magnetic field is shown schematically by following the arrows in Fig. 29. Another method of demagnetizing a part is to heat it above its Curie temperature (about 550°C for iron), where all residual magnetism disappears. This last process is the best means of removing all residual magnetism, but it does require the expense and time of an elevated heat treatment.

8 CONSIDERATIONS FOR INSPECTABILITY IN MATERIALS SELECTION

The reader will note that reliability of the inspection process is a very important parameter. This is so important that the description of any NDI procedure is incomplete without a thorough evaluation of its reliability. For this reason a special section of this chapter is devoted to inspection reliability. One might be tempted to assume that inspection reliability is related to the specific inspection method, but this is unfortunately not true. The reliability of each inspection process is a function of the technique used, the material, and its structural or geometric configuration, as well as the methodology of interpreting the data.

Considerations for inspectability in materials selection ranges from visual inspection[6] for esthetic applications to sophisticated inspection by a variety of nondestructive methods and processes.[8,10–12,14,16–19,21] The use of a material is a primary consideration in establishing requirements for inspection. Materials are initially selected on the basis of their properties. Much of the realization of modern technology implementation is due to increased reliability and confidence level in the ‘‘fitness for purpose’’ of the materials, components, structures, and assemblies. A significant factor in those increased is the use and improvements in nondestructive inspection capabilities and confidence level.

8.1 Confidence in Materials Properties

For engineering applications, expectations and confidence that materials can be produced with consistent properties is essential to the reliability and confidence level in the end product. Process control in producing materials is required to assure that consistent properties are produced and inspection may be an essential element in both process control and final product acceptance. For example, heat treatment of structural materials is often monitored by eddy current conductivity and indentation hardness measurements to provide assurance that desired properties have been produced.

In some cases, the practical use of an excellent engineering material may be critically dependent on in-process application of nondestructive testing procedures to detect and remove anomalies that are inherent to the materials production process. An example is the X-radiography of titanium sponge material to detect and remove tungsten inclusions that may be introduced during the melting and alloying process.

8.2 Structural Integrity

The structural integrity of engineering materials, components, assemblies, systems, and structures is also a primary element in successful designs and applications. Historically, structural integrity was provided by judgment and experience in initial materials selection, and assurance was provided by a combination of experience in process control and production and a safety margin to reduce risk of failure. A design was often proof tested to validate its structural integrity, and subsequent units were assumed to be identical to the validated component, and variance in properties was assumed to be accounted for by the design margin. An article was said to be ‘‘proof tested’’ if it was used in an environment that was sufficiently extreme for some defined period of time. The extreme conditions were defined to be more severe than any that the component could be reasonably expected to see in service.

The companion consideration in assuring structural integrity is to provide an alternate load path such that the load/function of the failed element is carried by companion structure until shutdown/maintenance/repair can be effected. ‘‘Fail-safe’’ design is always a basic consideration but is not always possible for primary structure such as pressure vessels.

Safe-Life Design

The structural/performance integrity of a system for a ‘‘safe life’’ was historically assured by a combination of experience (prior art), design margin, subelement tests, and full-scale ‘‘fatigue test articles.’’ Test components were assumed to be either ‘‘flaw free’’ or to contain small (but undetectable) flaws of an assumed size at the time of production, and flaw growth was calculated from that assumed small flaw size. Crack growth rate and life were based on materials properties testing (fatigue life). Various measures were used to assure that the production components were ‘‘identical’’ to the test article. These included the application of nondestructive inspection methods and procedures.

Periodic Inspection and Maintenance

Continuing confidence in structural integrity is provided by periodic (often calendar date programmed) inspection and maintenance to address undetected variances in production, design short falls, functional use, and environmentally induced anomalies. The design and application risks were once unquantified but were driven to conservatism by experience in applications (engineering art).

Technology Advancement

The historic methods of design and life-cycle management were successful in providing functionality and an unquantified confidence level in inherent and continuing structural integrity. In like manner, the inspection methods applied to such designs were based on experience, and the availability of inspection method(s), which provided a perceived confidence in design. These are the principal elements of the experience-based design/decision process. The advent and development of fracture mechanics as an engineering tool offers a method of quantifying the effects of material flaws on structural integrity. A key factor in application is detection and quantification of anomalies (primarily cracks) throughout the component’s service life.

Damage-Tolerant Design

Fracture mechanics provides a method of predicting the capability of material to perform in a specific service environment with a crack of a given size under a given load (environment, etc.). It also defines a new material property termed fracture toughness, which is roughly the ability of a material to resist the rapid growth of a crack from a small size to failure of the material. Quantification of crack detection capability and sizing have new requirements for nondestructive inspection and life-cycle management with corresponding increases in design efficiency. High efficiency, high confidence, and reduced life-cycle cost benefits from damage-tolerant design generated a new scenario for materials selection and design usage.

Use of fracture mechanics as a design tool significantly changes the design process by replacing a part of the engineering art (assumptions of material flaw content at the time of component production and considerations of flaw behavior in various environments) with quantifiable engineering parameters. Furthermore, the design life and life-cycle maintenance can now be quantified using new design parameters.

Static Considerations

A typical design sequence includes:

1. Selection of a structural material
2. Calculation of the maximum service load
3. Calculation of a "critical crack size" that could result in fracture using fracture mechanic design tools

Figure 30 shows a simple case relating the critical crack size in a selected material at various loads. If the calculated critical crack size is very large (low-stress level) in the selected application, the design process may proceed with other considerations. Useful stress levels often require consideration for small cracks and a method of verifying structural integrity may be in order. In a traditional design, proof test was often used. Proof testing has many advantages and is often used in conjunction with other verification methods. In some cases, the crack size that may be successfully screened by proof testing is larger than that screened by nondestructive evaluation (NDE) methods, and a larger design margin may be attained by use of inspection methods. This advantage is shown schematically in Fig. 30.

Dynamic Considerations

The next step in the design process is to consider growth of a crack during the service life of the design component. At a given load (environment, etc.), crack

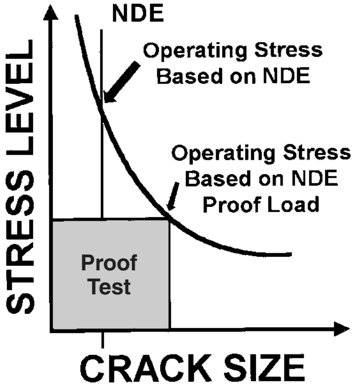

Fig. 30 Crack size as a function of stress level.

growth rate may be calculated and a safe life predicted by assuring that a preexisting crack (screened on entry into service) does not grow to a critical size during the service life. A part of that safe-life assurance may include scheduled maintenance/inspections at various service cycles to remove cracks that could grow to a critical size before the next scheduled maintenance inspection point.

A design logic sequence incorporating considerations for fracture control is shown in Fig. 31. Special designation of fracture-critical components is often required on engineering drawings. Notes may include requirements for special handling, environmental control, special inspection methods, or special testing methods. Special inspection methods include quantified crack detection capability assessment and validation.

8.3 Benefits of Quantified Crack Detection

In design and life-cycle management considerations, the benefits of quantified crack detection capabilities are multiplied in both risk analysis/reduction and in life-cycle cost of ownership. These benefits are manifested in:

Material Quality Selection

The selection of a material quality/grade for a given application is often driven by the availability and/or cost of inspection that is required to provide the required confidence level for use. For example, the grade of titanium used in rotating gas turbine engine (jet) components is considerably different from that used in a submarine hull. The material quality or grade is often determined by the defect size that can be detected with confidence in that material.

Impact on Structural Integrity

Damage tolerance is based on the ability of a component, system or structure to maintain structural integrity in the presence of an assumed included flaw of

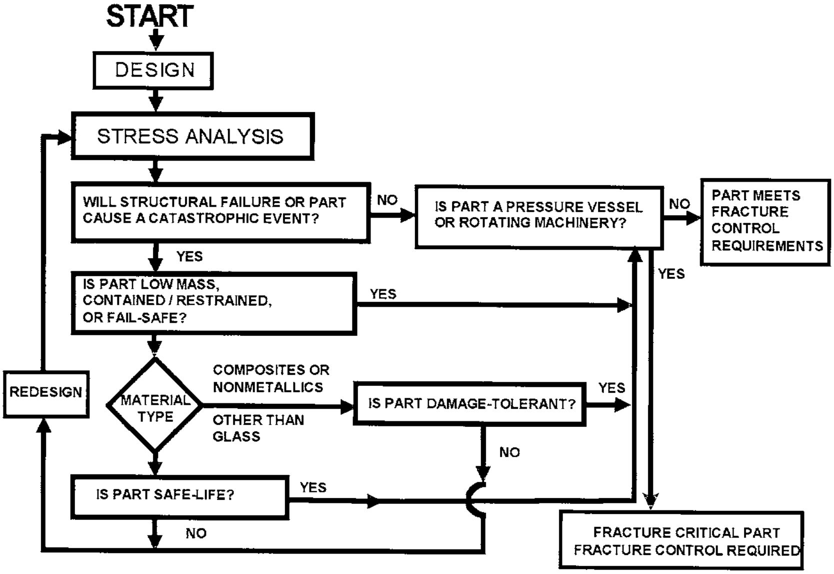

Fig. 31 Fracture control plan logic.

a given size. The design efficiency (load levels), margin, etc. are based in part on the flaw size, which can be detected prior to entry into service and at various programmed inspection maintenance intervals throughout the service life.

Inspection and Maintenance

Inspection and maintenance requirements for a given design/application can be quantified and periodic maintenance can be scheduled on the basis of design requirements and use rather than on an arbitrary calendar schedule. Additional confidence/safety margin can be incorporated and maintenance can be adjusted to account for design short falls, unanticipated service usage, unanticipated environmental degradation, and risk reduction. The economics of quantifying inspection, maintenance, and overhaul may drive incorporation of damage tolerance design/analysis to traditional applications where tradition and historical confidence have provided high confidence in structural integrity in past applications.

Safe Life

The ability to quantify safe life for an engineering structure also has tremendous economic impact. When the initial assumed flaw size is quantified, the time for that flaw to grow to a critical size may be quantified based on fatigue life and design load analyses. In many cases, the initial cost of application may be much lower than the life-cycle cost of ownership. Safe-life analyses provides a capability to provide better selection of material for both structural/functional integrity and for cost of ownership during that life.

Life Extension

Demands continually increase for life extension of engineering systems and structures and systems. Indeed many commercial aircraft in service are being used well beyond their design life. Within limits, service life extension can be justified by analysis when the basis for the design is quantified and the life-cycle inspection/maintenance cycles are quantified. Life extension may be effected with no change in risk or confidence level by consideration of fatigue life behavior and the incorporation of quantified inspection procedures.

Life Extensions Considerations. Consider the initiation and growth of a flaw in service. Service life will include nucleation and stable flaw growth until it reaches a critical size and precipitates fracture. This process is shown schematically in Fig. 32. Failure can be avoided by detection of that flaw before it grows to a critical size. The interaction of inspection is thus to apply an inspection method that has the capability to detect flaws before they grow to a critical size.

Material behavior is such that there is a significant variance between the time that a fatigue crack initiates for a given material lot and the time when the all components will initiate a flaw. If inspection is applied, those components that have early flaw initiation may be removed from service and the remainder of the lot may continue in service with extended life. Figure 33 shows a typical spread in crack initiation for a material lot in service and periodic inspections to remove those components that have initiated a flaw.

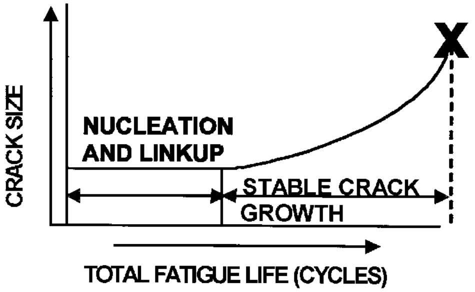

Fig. 32 Crack nucleation and growth to fracture.

Structural integrity can be assured by periodic inspection to remove (replace/repair) components that have developed cracks and thereby provide a quantified continuing safe life. The inspection/maintenance interval is a function of the service life and the capability of nondestructive inspection capabilities to reliably detect components with cracks or cracklike flaws.

Retirement for Cause. The concept of "retirement for cause" (RFC) is to discard a component when the probability of its failure reaches unacceptable levels. This scenario has enabled both knowledge-based service life extension and significant cost savings for many modern engineering systems and components. The periodic inspection and removal of flawed components concept is shown schematically in Fig. 34.

The RFC philosophy means that a structure or fleet of structures can be managed by knowledge of crack initiation and growth characteristics. The philosophy assumes that components that contain small cracks, at some size level, have been removed at the time of production acceptance. Components/structures that do enter service are subjected to cyclic loading (fatigue) during their service lifetime. The mean time to initiation of a fatigue crack in service varies widely, as shown in Fig. 33. As the components age in service, a few will be expected to initiate and grow fatigue cracks earlier than the general population. Inspection is then performed on the group of similar aged structures or components, and those that contain size cracks above a certain size are removed from service.

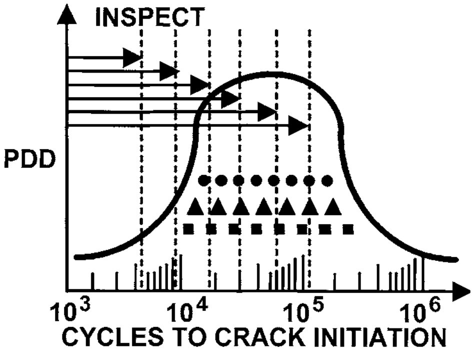

Fig. 33 Life extension by inspection and removal of cracks that initiate due to fatigue.

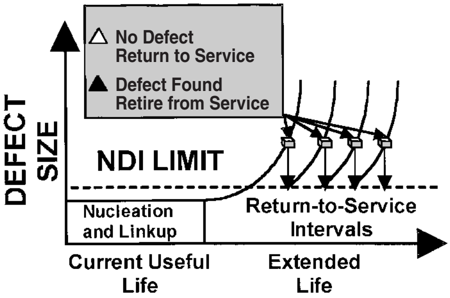

Fig. 34 Retirement for cause life-cycle maintenance incorporating quantitative inspection.

The remaining components are returned to service and continue in useful life to the next calculated inspection interval. The same group is reinspected at the next interval, and those containing cracks above a certain size are removed with the remainder again returned to service. By continuing this life-cycle management process, the maximum useful life of all the components or fleet of structures may be realized. The key to this process is quantification of the inspection process to determine the flaw size that can be reliably detected and removed from service. The calculated inspection interval is based on probable growth of any detected flaws before the next inspection cycle. As an added safety factor, the inspection interval is usually set at one-third to one-half of the calculated time required for a flaw to grow to a critical failure inducing size. In this manner, structural integrity can be maintained and service life can be extended until the component is retired for cause—crack initiation, wear, corrosion, dimensional changes, etc.

Damage-tolerant design and life-cycle management are dependent on detection and removal of cracks and cracklike stress risers during production and during life. Such detection must necessarily be nondestructive and quantification of detection capabilities are required.

Requirements for Quantitative Nondestructive Inspection

Nondestructive inspections/evaluations (NDI/E) have traditionally been routinely applied to a variety of engineering materials, components, structures, and systems to provide confidence in structural integrity. Development and implementation of NDI/E methods were often prompted by a catastrophic failure of an important engineering system. This experience base of previous failures was integrated into "engineering art," and NDI/E provided a deterministic but unquantified measure of confidence in structural integrity and continuing service. The advent and implementation of fracture mechanics in engineering design established the need for quantification of the capabilities of applied NDI/E procedures. That need moved nondestructive inspection requirements from a tradesman's art to an integrated engineering technology.

Requirements to establish a reliable nondestructive inspection include the following:

1. *Access.* Access to perform an inspection is essential and must be considered as a primary engineering design parameter. An inspection cannot be performed unless key areas of the component are accessible. This may require disassembly in some cases and must be considered as a part of the life-cycle maintenance procedures.
2. *Capability.* The inspection method must be capable of detecting the required flaw sizes to a defined reliability and confidence levels. Selection of the applicable inspection method is primarily the responsibility of the NDI engineer. A design cannot be supported if the critical flaw size is below the detection capability level.
3. *Reliable.* The reliability of an inspection method is dependent, in part, on the reproducibility of the measurements being made by the NDI procedure. The reproducibility of a procedure is, in turn, largely dependent on the "calibration"/setup used for the NDI procedure. Additionally, the process must be repeatable, i.e., the NDI procedure is dependent on process control rigor in application of the NDI method/process.
4. *Human Factors.* Since the reproducibility and repeatability of an NDI procedure are dependent on rigor in process control, the skill of the human operator is an important factor in NDI application. Most NDI methods/procedures require dexterity in application and detection/pattern recognition skills for "interpretation" and decision processing. Most NDI methods produce indirect measurements of a property or configuration and relates those measurement to a desired property or behavior. Proficiency in such relationships are a product of the knowledge and skill of the NDI engineer and the knowledge and skill of the inspector/operator.

8.4 Quantification of NDE Capabilities

Nondestructive inspection methods have a long history of application that range from simple visual inspections to complex ultrasonic and eddy current inspections. Since most inspections were originally practiced as an art, the craft was subject to a wide range of assumptions, exaggerations, misinformation, and misapplications. The requirement to quantify the detection capability has reduced many of those deficiencies but has generated new barriers from practitioners who resist change. Without discipline and quantification, many of inspection procedures must be regarded as "inspection ceremonies." This applies to many established procedures that were generated and perpetuated by "specification." The issue at hand is not *"the smallest flaw that has been detected, but the largest flaw that has been missed"* by an inspection procedure or operator.

The methodology that is applied to quantification of nondestructive inspection/evaluation capability is known as a "probability of detection (POD)" assessment. The single-valued inspection capability parameter that is provided by the POD method is a crack size that can be detected (reliably) by application of an inspection procedure. That value is often quoted as the 90/95 or the a_{NDE} (nondestructive evaluation) capability/parameter and is the value that is used in engineering design for use of nondestructive inspection as an element of validating component structural integrity.

8.5 Probability of Detection

The probability of detection (POD) methodology was generated to provide some of the confidence level in flaw detection capability that is used in engineering design. The basic guidelines and values for materials properties are available from MIL-HDBK-5.[67] The number of parameters and variances inherent to the application of an inspection procedure greatly exceed the materials properties variances, and the POD method is intended to account for the end-to-end variances and to provide a detection capability as a function of flaw size. Figure 35 is an example of a typical POD curve. The a_{NDE} value is the point at which the POD curve crosses the 90% POD threshold. This point has been selected by convention and is the basis for most modern design applications.

A POD curve is generated by passing a large number of flaws of varying size through an inspection process/procedure and recording either the "hit/miss" (detection or nondetection) data or the scalar output of the procedure. The data are then fit to a mathematical model[68] to provide rigor and consistency in the analysis and to produce the POD result. Typically 60 or more cracks of varying sizes are required to produce the POD curve.[69]

The POD method is specific to the flaw/artifact variables, inspection method, the inspection procedure, the inspection equipment, the inspection process, calibration, detection threshold used in the decision processing, test coupon/object variables, geometry, inspection environment, and human factors and is thus an end-to-end process measurement value. If any of the parameters change, the POD process must be repeated for rigor in application. The importance of process control in actual implementation is self-evident. Generation of a POD curve is expensive. Methods of reducing cost are constant considerations. In engineering application of the deterministic parameters, reference data are often used as the starting point for analysis. The *NDE Capabilities Data Book*[70] provides a handbook reference of POD curves that have been generated for various materials, inspection processes, and inspection conditions. This reference is a starting point when considering incorporation of an inspection requirement into a design.

For damage-tolerant designs, it is important to consider the capabilities of various nondestructive inspection methods for both purposes of cost of the in-

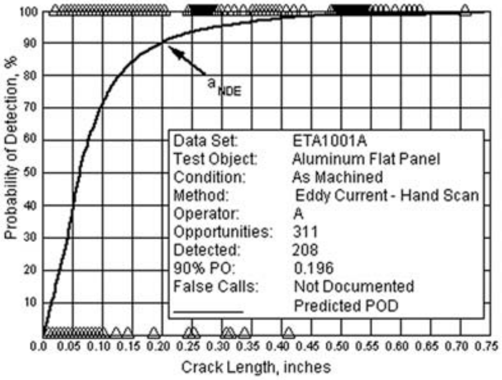

Fig. 35

spection and the cost/benefits ratio of inspection capabilities on life-cycle management. For example, fluorescent penetrant inspection is a widely used inspection method due to its inherent capabilities, wide area coverage, and economical cost of application. In general application, the capability of fluorescent penetrant processes is the detection of 0.100- to 0.150-in. long cracks. Fluorescent penetrant inspection is capable of detection cracks smaller than 0.100–0.150 in. in size but requires more discipline and control with corresponding increase in application cost. The lower limit for practical penetrant inspection is 0.030 in. This level requires extraordinary process control and discipline and is therefore not applicable to most components. If detection of smaller cracks than this is required, then it is necessary to apply alternate inspection methods such as eddy current. Eddy current inspection is in turn a focused inspection method and therefore has a higher cost. Regardless of the NDI method selected, it may be necessary to assess and demonstrate detection capability using the POD method (for fracture-critical applications). The demonstrated capability is specific to a facility, equipment, procedure, and operator. Only operators so qualified may be allowed to perform inspections of fracture-critical components.

False confidence in crack detection capabilities is the norm for many inspection facilities and operations. Since crack-to-crack variance is one of the POD process parameters, small cracks may be detected and larger cracks missed. Unfortunately, statistical variance in “calibration” and process control is often a cause for variance in detection. This fact reinforces the requirement to assure a stable NDI procedure (calibration and process control) before an assessment by the POD method is initiated.

The POD method of assessment has proven useful, not only as tool to meet design engineering requirements but also as a tool for a quantifiable comparison of the performance of various NDI procedures, to qualify inspection facilities and personnel, and to validate critical procedures in difficult inspection applications.

Lessons Learned from POD Assessments

A large number of POD assessments have been completed and results documented in various applications. The primary requirement is to have a stable calibration and process controls before a POD assessment is initiated. Rigid calibration and process controls are necessary in application to provide confidence that the same level of detection has been maintained. Reliable application demands reproducibility and repeatability to maintain a capability. The POD process is sensitive to a wide range of parameters. These include:

- Flaw (artifact) variables
- Test object variables
- Inspection method variables
- Materials variables
- Equipment variables
- Inspection procedure variables
- Inspection process variables
- Calibration variables

- Acceptance criteria/decision variables
- Human factors

Individual NDI procedures and applications considerations and identifiable sources of variance supplement this list. Variance in a single parameter will vary the POD and thus the capability of the NDI procedure.

8.6 General Process Control Is Required in All NDI Applications

Inspection for fracture control is a specialized application and reflects the full capabilities and benefits of quantitative NDI. Most engineering components are not fracture critical and do not require the rigor of demonstrating flaw detection capabilities. Rigorous quality control in these applications is, however, required due to the dependence of NDI capabilities on general structural integrity and life management. Cost should not be an issue since it costs no more to perform in an undisciplined (unreliable) manner than it does to perform in a discipline (reliable) manner. The labor may cost a small amount more, but the materials and equipment are the same. The results can vary significantly in terms of legal and economic exposure.

Care must be exercised in establishing engineering requirements to assure that the required level of detection is applied. Specifying a small flaw size does not necessarily provide a superior or applicable inspection, but may add unnecessarily to the inspection cost. A common practice in the past has been to specify ‘‘no defects/no cracks/or no flaws.’’ The practice arose for fracture-critical components where the engineer wanted to know about every flaw detected and thus be able to review and disposition all flaws found. This is an unacceptable practice since it leaves the level of inspection to the NDI engineer and no qualitative value is provided. An acceptable practice is to specify a critical flaw size and a reportable flaw size. This imposes the requirement to demonstrate detection capability at the reportable flaw size level and to reject at the critical flaw size level. This method provides a quantified margin for the inspection process and provides a higher confidence in results. The reportable flaw size level is especially useful for automated inspection processes since it provides a mechanism for reassessment and trend monitoring of inspection data for purposes of component system performance assessments. The melding of engineering design and NDI to provide quantitative analysis, specification, measurement, and reporting has supported engineering efficiency and confidence levels that are the hallmark of modern engineering materials, components, structures, and systems.

9 SUMMARY

The intent of this chapter is to provide the reader with a basic understanding of the fundamentals of nondestructive inspection both from the fundamental science and reliability standpoints. This latter point is important since the design process often requires the engineer to specify a reliability of a component. While all designers realize that a material or manufacturing process specification significantly affects the reliability of a design, the inclusion of an inspection procedure is also a significant factor in reliability. In choosing a particular material, the designer automatically limits the inspection choices. For example, only ferromagnetic alloys such as cast iron and most steels are inspectable with magnetic particle inspection. Another example might be the limited ability to detect deeply buried flaws using thermal inspection methods on highly conductive aluminum components. Therefore, the design engineer should be cognizant of the limitations that material choice places on the applicable inspection methods. In these days of product liability law suits, an understanding of inspection technology has become another critical component of the designer's required skill set. The authors hope that this chapter provides a first step to acquainting design and process engineers with some of the basic considerations necessary to achieve reliable, cost-effective products. If additional information is needed, the reader is referred to the references or to Internet sites cited.

APPENDIX A: ULTRASONIC PROPERTIES OF COMMON MATERIALS

Liquids

Liquid (20°C unless noted)	Longitudinal Wave Speed ($\times 10^5$ cm/s)	Density (g/cm^3)
Acetic acid	1.173	1.049
Acetone	1.192	0.792
Amyl acetate (26°C)	1.168	0.879
Aniline	0.656	1.022
Benzene	1.326	0.879
Blood (horse) (37°C)	1.571	—
Bromoform	0.928	2.890
n-Buytl alcohol	1.268	0.810
Caprylic acid	1.331	0.910
Carbon disulfide	1.158	1.263
Carbon tetrachloride	0.938	1.595
Cloroform	1.005	1.498
Formaldehyde (25°C)	1.587	0.815
Gasoline (34°C)	1.25	0.803
Glycerin	1.923	1.261
Kerosene (25°C)	1.315	0.82
Mercury	1.451	13.546
Methyl alcohol	1.123	0.796
Oils		
Campor (25°C)	1.390	—
Castor	1.500	0.969
Condenser	1.432	—
Olive (22°C)	1.440	0.918
SAE 20	1.74	0.87
Sperm (32°C)	1.411	—
Transformer	1.38	0.92
Oleic acid	1.333	0.873
n-Pentane	1.044	—
Silicon tetrachloride (30°C)	0.766	1.483
Toluene	1.328	0.67
Water (distilled)	1.482	1.00
m-Xylene	1.340	0.864

Solids: Metals

Metal (20°C unless noted)	Longitudinal Wave Speed ($\times 10^5$ cm/s)	Transverse Wave Speed ($\times 10^5$ cm/s)	Density (g/cm^3)
Aluminum			
Al (1100)	6.31	3.08	2.71
Al (2014)	6.37	3.07	2.80
Al (2024-T4)	6.37	3.16	2.77
Al (2117-T4)	6.50	3.12	2.80
Al (6061-T6)	6.31	3.14	2.70
Bearing babbit	2.30	—	10.1
Beryllium	12.890	8.880	1.82
Bismuth	2.18	1.10	9.80
Brass (70% Cu & 30% Zn)	4.37	2.10	8.50
Brass (naval)	4.43	2.12	8.42
Bronze (5% P)	3.53	2.32	8.86
Cadmium	2.78	1.50	8.64
Cerium	2.424	1.415	6.77
Chromium	6.608	4.005	7.20
Cobalt	5.88	3.10	8.90
Columbium	4.92	2.10	8.57
Constantan	5.177	2.625	8.88
Copper	4.759	2.325	8.93
Copper (110)	4.70	2.26	8.9
Dysprosium	2.296	1.733	8.53
Erbium	2.064	1.807	9.06
Europium	1.931	1.237	5.17
Gadolinium	2.927	1.677	7.89
Germanium	5.18	3.10	5.47
Gold	3.24	1.20	19.32
Hafnium	2.84	—	13.3
Hastelloy X	5.79	2.74	8.23
Hastelloy C	5.84	2.90	8.94
Holmium	3.089	1.729	8.80
Indium	2.56	0.74	7.30
Invar	4.657	2.658	—
Lanthanum	2.362	1.486	6.16
Lead	2.160	0.700	11.34
Lead (96% Pb & 6% Sb)	2.16	0.81	10.88
Lutetium	2.765	1.574	9.85
Magnesium	5.823	3.163	1.74
AM-35	5.79	3.10	1.74
FS-1	5.47	3.03	1.69
J-1	5.67	3.01	1.70
M1A	5.74	3.10	1.76
O-1	5.80	3.04	1.72
Manganese	4.66	2.35	7.39
Manganin	4.66	2.35	8.40

Metal (20°C unless noted)	Longitudinal Wave Speed ($\times 10^5$ cm/s)	Transverse Wave Speed ($\times 10^5$ cm/s)	Density (g/cm^3)
Molybdenum	6.29	3.35	10.2
Nickel			
Pure	5.63	2.96	8.88
Inconel	5.82	3.02	8.5
Inconel (X-750)	5.94	3.12	8.3
Inconel (wrought)	7.82	3.02	8.25
Monel	5.35	2.72	8.83
Monel (wrought)	6.02	2.72	8.83
Silver–nickel (18%)	4.62	2.32	8.75
German silver	4.76	2.16	8.40
Neodymium	2.751	1.502	7.10
Platinum	3.96	1.67	21.4
Potassium	2.47	1.22	0.862
Praseodymium	2.639	1.437	6.75
Samarium	2.875	1.618	7.48
Silver	3.60	1.59	10.5
Sodium	3.03	1.70	0.97
Steel			
1020	5.89	3.24	7.71
1095	5.90	3.19	7.80
4150, Rc14	5.86	2.79	7.84
4150, Rc 18	5.88	3.18	7.82
4150, Rc 43	5.87	3.20	7.81
4150, Rc 64	5.83	2.77	7.80
4340	5.85	3.24	7.80
52100 Annealed	5.99	3.27	7.83
52100 Hardened	5.89	3.20	7.8
D6 Tool Steel Annealed	6.14	3.31	7.7
Stainless Steels			
302	5.66	3.12	7.9
304L	5.64	3.07	7.9
347	5.74	3.10	7.91
410	5.39	2.99	7.67
430	6.01	3.36	7.7
Tantalum	4.10	2.90	16.6
Thorium	2.94	1.56	11.3
Thulium	3.009	1.809	9.29
Tin	3.32	1.67	7.29
Titanium (Ti-6-4)	6.18	3.29	4.50
Tungsten			
Annealed	5.221	2.887	19.25
Drawn	5.410	2.640	19.25
Uranium	3.37	1.98	18.7
Vanadium	6.023	2.774	6.03
Ytterbium	1.946	1.193	6.99
Yttrium	4.10	2.38	4.34
Zinc	4.187	2.421	7.10
Zirconium	4.65	2.25	6.48

Solids: Ceramics

Ceramic (20°C unless noted)	Longitudinal Wave Speed ($\times 10^5$ cm/s)	Transverse Wave Speed ($\times 10^5$ cm/s)	Density (g/cm^3)
Aluminum oxide	10.84	6.36	3.98
Barium nitrate	4.12	2.28	3.24
Barium titanate	5.65	3.03	5.5
Bone (human tibia)	4.00	1.97	1.7–2.0
Cobalt oxide	6.56	3.32	6.39
Concrete	4.25–5.25	—	2.60
Glass			
Crown	5.66	3.42	2.50
Flint	4.26	2.56	3.60
Lead	3.76	2.22	4.6
Plate	5.77	3.43	2.51
Pyrex	5.57	3.44	2.23
Soft	5.40	—	2.40
Granite	3.95	—	2.75
Graphite	4.21	2.03	2.25
Ice (−16°C)	3.83	1.92	0.94
Indium antimonide	3.59	1.91	—
Lead nitrate	3.28	1.47	4.53
Lithium fluoride	6.56	3.84	2.64
Magnesium oxide	9.32	5.76	3.58
Manganese oxide	6.68	3.59	5.37
Nickel oxide	6.60	3.68	6.79
Porcelain	5.34	3.12	2.41
Quartz			
Crystalline	5.73	—	2.65
Fused	5.57	3.52	2.60
Polycrystalline	5.75	3.72	2.65
Rock salt	4.60	2.71	2.17
Titanium dioxide (Rutile)	8.72	4.44	4.26
Sandstone	2.92	1.84	2.2–2.4
Sapphire (c-axis)	11.91	7.66	3.97
Slate	4.50	—	2.6–3.3
Titanium carbide	8.27	5.16	5.15
Tourmaline (Z-cut)	7.54	—	3.10
Tungsten carbide	6.66	3.98	10.15
Yttrium iron garnet	7.29	4.41	5.17
Zinc sulfide	5.17	2.42	4.02
Zinc oxide	6.00	2.84	5.61

Solids: Polymers

Polymer (20°C Unless Noted)	Longitudinal Wave Speed ($\times 10^5$ cm/s)	Transverse Wave Speed ($\times 10^5$ cm/s)	Density (g/cm^3)
Acrylic resin	2.67	1.12	1.18
Bakelite	2.59	—	1.40
Buytl rubber	1.99	—	1.13
Cellulose acetate	2.45	—	1.30

Polymer (20°C Unless Noted)	Longitudinal Wave Speed ($\times 10^5$ cm/s)	Transverse Wave Speed ($\times 10^5$ cm/s)	Density (g/cm^3)
Cork	0.5	—	0.2
Delrin (acetalhomo polymer) (0°C)	2.515	—	1.42
Ebonite	2.50	—	1.15
Lexan (Polycarbonate 0°C)	2.28	—	1.19
Neoprene	1.730	—	1.42
Nylon	2.68	—	—
Nylon 6,6	1.68	—	—
Paraffin	2.20	0.83	—
Perspex	2.70	1.33	1.29
Phenolic	1.42	—	1.34
Plexiglas			
UVA	2.76	—	1.27
UVA II	2.73	1.43	1.18
Polyacrylonitrile–butadiene–styrene I	2.16	1.43	1.18
Polyacrylonitrile–butadiene–styrene II	2.20	0.810	1.022
Polybutadiene rubber	1.57	—	1.10
Polycaprolactam	2.700	1.12	1.146
Polycarboranesilonane	1.450	—	1.041
Polydimethylsiloxane	1.020	—	1.045
Polyepoxide + glass spheres I	2.220	1.170	0.691
Polyepoxide + glass spheres II	2.400	1.280	0.718
Polyepoxide + glass spheres III	2.100	1.020	0.793
Polyepoxide + MPDA	2.820	1.230	1.205
Polyester + water	1.840	0.650	1.042
Polyethylene	2.67	—	1.10
Polyhexamethylene adipamide	2.710	1.120	1.147
Polymethacrylate	2.690	1.344	1.191
Polyoxymethylene	2.440	1.000	1.425
Polyproplene	2.650	1.300	0.913
Polystyrene	2.400	1.150	1.052
Polysulfane resin	2.297	—	1.24
Polytetrafluoroethylene (Teflon)	1.380	—	2.177
Polyvinylbutyral	2.350	—	1.107
Polyvinyl chloride	2.300	—	—
Polyvinylidene chloride	2.400	—	—
Polyvinylidene fluoride	1.930	—	1.779
Rubber			
India	1.48	—	0.90
Natural	1.55	—	1.12
Rubber/carbon (100/40)	1.68	—	—
Silicon rubber	0.948	—	1.48

APPENDIX B: ELECTRICAL RESISTIVITIES AND CONDUCTIVITIES OF METALS AND ALLOYS[17]

Common Name (Classification)	Resistivity (Ω-cm $\times 10^{-6}$)	Conductivity (% IACS)
Ingot iron (included for comparison)	9	19
Plain carbon steel (AISI–SAE 1020)	10	17
Stainless steel type 304	72	2.4
Cast gray iron (ASTH A48-48, Class 25)	67	2.6
Malleable iron (ASTM A47)	30	5′7
Ductile cast iron (ASTH A339, A395)	60	2.9
Ni (resist cast iron, type 2)	170	1.0
Cast 28-7 alloy 11D (ASTH A297–63T)	41	4.2
Hastelloy C	139	1.2
Hastelloy X	115	1.5
Haynes stellite alloy 25	8B	2.0
Inconel X (annealed)	122	1.4
Inconel 600	98	1.7
Aluminum alloy 3003, rolled (ASTH B221)	4	43
Aluminum alloy 2017, annealed (ASTH B221)	4	43
Aluminum alloy 380 (ASTH SC84B)	7.5	23
A# Aluminum alloy		
6061-T-6	4.1	42
7075-T-6	5.3	32
2024-T-4	5.2	30
Copper (ASTH B152, B124, B133, B1, B2, B3)	1.7	1.0×10^2
Yellow brass or high brass (ASTH B36, B134, B135)	7	25
70-30 brass	6.2	28
Aluminum bronze		
ASTH B169, alloy A	12	14
ASTH B124, B130		
Phosphor bronzes	16	11
Nickel silver I B% alloy A wrought (ASTH B 122, No. 2)	29	5.9
Cupronickel 30%	35	4.9
Red brass, cast (ASTH B30, No. 4A)	11	16
Chemical lead	21	8.2
Antimonial lead (hard lead)	23	7.5
Solder 50-50	15	N
Ti–6Al–4V alloy	172	1.0
Magnesium alloy AZ31 B	9	19
K Monel	58	3.0
Nickel (ASTH B160, B161, B162)	10	17
Cupronickel 55-45 (Constantan)	49	3.5
Commercial titanium	80	2.2
Waspaloy	123	1.4
Zinc (ASTH B69)	6	29
Zircaloy-2	72	2.4
Zirconium (commercial)	41	4.2

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PART 5

FAILURE ANALYSIS

Handbook of Materials Selection. Edited by Myer Kutz

CHAPTER 25

FAILURE MODES: PERFORMANCE AND SERVICE REQUIREMENTS FOR METALS

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1 CRITERIA OF FAILURE

Any change in the size, shape, or material properties of a structure, machine, or machine part that renders it incapable of performing its intended function must be regarded as a mechanical failure of the device. It should be carefully noted that the key concept here is that *improper functioning* of a machine part constitutes failure. Thus, a shear pin that does *not* separate into two or more pieces upon the application of a preselected overload must be regarded as having failed

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as surely as a drive shaft has failed if it *does* separate into two pieces under normal expected operating loads.

Failure of a device or structure to function properly might be brought about by any one or a combination of many different responses to loads and environments while in service. For example, too much or too little elastic deformation might produce failure. A fractured load-carrying structural member or a shear pin that does not shear under overload conditions each would constitute failure. Progression of a crack due to fluctuating loads or aggressive environment might lead to failure after a period of time if resulting excessive deflection or fracture interferes with proper machine function.

A primary responsibility of any mechanical designer is to ensure that his or her design functions as intended for the prescribed design lifetime and, at the same time, that it be competitive in the marketplace. Success in designing competitive products while averting premature mechanical failures can be achieved consistently only by recognizing and evaluating all potential modes of failure that might govern the design. To recognize potential failure modes a designer must be acquainted with the array of failure modes observed in practice, and with the conditions leading to these failures. The following section summarizes the mechanical failure modes most commonly observed in practice, followed by a brief description of each one.

2 FAILURE MODES

A failure mode may be defined as the physical process or processes that take place or that combine their effects to produce a failure. In the following list of commonly observed failure modes, it may be noted that some failure modes are unilateral phenomena, whereas others are combined phenomena. For example, fatigue is listed as a failure mode, corrosion is listed as a failure mode, and corrosion fatigue is listed as still another failure mode. Such combinations are included because they are commonly observed, important, and often *synergistic*. In the case of corrosion fatigue, for example, the presence of active corrosion aggravates the fatigue process and at the same time the presence of a fluctuating load accelerates the corrosion process.

The following list is not presented in any special order, but it includes all commonly observed modes of mechanical failure[1]:

1. Force and/or temperature-induced elastic deformation
2. Yielding
3. Brinnelling
4. Ductile rupture
5. Brittle fracture
6. Fatigue
    a. High-cycle fatigue
    b. Low-cycle fatigue
    c. Thermal fatigue
    d. Surface fatigue
    e. Impact fatigue

   - **f.** Corrosion fatigue
   - **g.** Fretting fatigue
7. Corrosion
   - **a.** Direct chemical attack
   - **b.** Galvanic corrosion
   - **c.** Crevice corrosion
   - **d.** Pitting corrosion
   - **e.** Intergranular corrosion
   - **f.** Selective leaching
   - **g.** Erosion corrosion
   - **h.** Cavitation corrosion
   - **i.** Hydrogen damage
   - **j.** Biological corrosion
   - **k.** Stress corrosion
8. Wear
   - **a.** Adhesive wear
   - **b.** Abrasive wear
   - **c.** Corrosive wear
   - **d.** Surface fatigue wear
   - **e.** Deformation wear
   - **f.** Impact wear
   - **g.** Fretting wear
9. Impact
   - **a.** Impact fracture
   - **b.** Impact deformation
   - **c.** Impact wear
   - **d.** Impact fretting
   - **e.** Impact fatigue
10. Fretting
    - **a.** Fretting fatigue
    - **b.** Fretting wear
    - **c.** Fretting corrosion
11. Creep
12. Thermal relaxation
13. Stress rupture
14. Thermal shock
15. Galling and seizure
16. Spalling
17. Radiation damage
18. Buckling
19. Creep buckling

20. Stress corrosion
21. Corrosion wear
22. Corrosion fatigue
23. Combined creep and fatigue

As commonly used in engineering practice, the failure modes just listed may be defined and described briefly as follows. It should be emphasized that these failure modes only produce failure when they generate a set of circumstances that interferes with the proper functioning of a machine or device.

Force and/or temperature-induced elastic deformation failure occurs whenever the elastic (recoverable) deformation in a machine member, induced by the imposed operational loads or temperatures, becomes large enough to interfere with the ability of the machine to perform its intended function satisfactorily.

Yielding failure occurs when the plastic (unrecoverable) deformation in a ductile machine member, brought about by the imposed operational loads or motions, becomes large enough to interfere with the ability of the machine to perform its intended function satisfactorily.

Brinnelling failure occurs when the static forces between two curved surfaces in contact result in local yielding of one or both mating members to produce a permanent surface discontinuity of significant size. For example, if a ball bearing is statically loaded so that a ball is forced to permanently indent the race through local plastic flow, the race is brinnelled. Subsequent operation of the bearing might result in intolerably increased vibration, noise, and heating; and, therefore, failure would have occurred.

Ductile rupture failure occurs when the plastic deformation, in a machine part that exhibits ductile behavior, is carried to the extreme so that the member separates into two pieces. Initiation and coalescence of internal voids slowly propagate to failure, leaving a dull, fibrous rupture surface.

Brittle fracture failure occurs when the elastic deformation, in a machine part that exhibits brittle behavior, is carried to the extreme so that the primary interatomic bonds are broken and the member separates into two or more pieces. Preexisting flaws or growing cracks form initiation sites for very rapid crack propagation to catastrophic failure, leaving a granular, multifaceted fracture surface.

Fatigue failure is a general term given to the sudden and catastrophic separation of a machine part into two or more pieces as a result of the application of fluctuating loads or deformations over a period of time. Failure takes place by the initiation and propagation of a crack until it becomes unstable and propagates suddenly to failure. The loads and deformations that typically cause failure by fatigue are far below the static or monotonic failure levels. When loads or deformations are of such magnitude that more than about 10,000 cycles are required to produce failure, the phenomenon is usually termed *high-cycle fatigue*. When loads or deformations are of such magnitude that less than about 10,000 cycles are required to produce failure, the phenomenon is usually termed *low-cycle fatigue*. When load or strain cycling is produced by a fluctuating temperature field in the machine part, the process is usually termed *thermal fatigue*. *Surface fatigue* failure, usually associated with rolling surfaces in contact, manifests itself as pitting, cracking, and spalling of the contacting surfaces as a result of the cyclic Hertz contact stresses that result in maximum values of cyclic shear stresses slightly below the surface. The cyclic subsurface shear stresses generate cracks that propagate to the contacting surface, dislodging particles in the process to produce surface pitting. This phenomenon is often viewed as a type of wear. Impact fatigue, corrosion fatigue, and fretting fatigue are described later.

Corrosion failure, a very broad term, implies that a machine part is rendered incapable of performing its intended function because of the undesired deterioration of the material as a result of chemical or electrochemical interaction with the environment. Corrosion often interacts with other failure modes such as wear or fatigue. The many forms of corrosion include the following. *Direct chemical attack,* perhaps the most common type of corrosion, involves corrosive attack of the surface of the machine part exposed to the corrosive media, more or less uniformly over the entire exposed surface. *Galvanic corrosion* is an accelerated electrochemical corrosion that occurs when two dissimilar metals in electrical contact are made part of a circuit completed by a connecting pool or film of electrolyte or corrosive medium, leading to current flow and ensuing corrosion. *Crevice corrosion* is the accelerated corrosion process highly localized within crevices, cracks, or joints where small volume regions of stagnant solution are trapped in contact with the corroding metal. *Pitting corrosion* is a very localized attack that leads to the development of an array of holes or pits that penetrate the metal. *Intergranular corrosion* is the localized attack occurring at grain boundaries of certain copper, chromium, nickel, aluminum, magnesium, and zinc alloys when they are improperly heat treated or welded. Formation of local galvanic cells that precipitate corrosion products at the grain boundaries seriously degrades the material strength because of the intergranular corrosive process. *Selective leaching* is a corrosion process in which one element of a solid alloy is removed, such as in dezincification of brass alloys or graphitization of gray cast irons. *Erosion corrosion* is the accelerated chemical attack that results when abrasive or viscid material flows past a containing surface, continuously baring fresh, unprotected material to the corrosive medium. *Cavitation corrosion* is the accelerated chemical corrosion that results when, because of differences in vapor pressure, certain bubbles and cavities within a fluid collapse adjacent to the pressure-vessel walls, causing particles of the surface to be expelled, baring fresh, unprotected surface to the corrosive medium. *Hydrogen damage,* while not considered to be a form of direct corrosion, is induced by corrosion. Hydrogen damage includes hydrogen blistering, hydrogen embrittlement, hydrogen attack, and decarburization. *Biological corrosion* is a corrosion process that results from the activity of living organisms, usually by virtue of their processes of food ingestion and waste elimination, in which the waste products are corrosive acids or hydroxides. *Stress corrosion,* an extremely important type of corrosion, is described separately later.

Wear is the undesired cumulative change in dimensions caused by the gradual removal of discrete particles from contacting surfaces in motion, usually sliding, predominantly as a result of mechanical action. Wear is not a single process, but a number of different processes that can take place by themselves or in combination, resulting in material removal from contacting surfaces through a complex combination of local shearing, plowing, gouging, welding, tearing, and others. *Adhesive wear* takes place because of high local pressure and welding at asperity contact sites, followed by motion-induced plastic deformation and rupture of asperity functions, with resulting metal removal or transfer. *Abrasive wear* takes place when the wear particles are removed from the surface by the plowing, gouging, and cutting action of the asperities of a harder mating surface or by hard particles entrapped between the mating surfaces. When the conditions for either adhesive wear or abrasive wear coexist with conditions that lead to corrosion, the processes interact synergistically to produce *corrosive wear*. As described earlier, *surface fatigue wear* is a wear phenomenon associated with curved surfaces in rolling or sliding contact, in which subsurface cyclic shear stresses initiate microcracks that propagate to the surface to spall out macroscopic particles and form wear pits. *Deformation wear* arises as a result of repeated *plastic* deformation at the wearing surfaces, producing a matrix of cracks that grow and coalesce to form wear particles. Deformation wear is often caused by severe impact loading. *Impact wear* is impact-induced repeated *elastic* deformation at the wearing surface that produces a matrix of cracks that grows in accordance with the surface fatigue description just given. Fretting wear is described later.

Impact failure results when a machine member is subjected to nonstatic loads that produce in the part stresses or deformations of such magnitude that the member no longer is capable of performing its function. The failure is brought about by the interaction of stress or strain waves generated by dynamic or suddenly applied loads, which may induce local stresses and strains many times greater than would be induced by the static application of the same loads. If the magnitudes of the stresses and strains are sufficiently high to cause separation into two or more parts, the failure is called *impact fracture*. If the impact produces intolerable elastic or plastic deformation, the resulting failure is called *impact deformation*. If repeated impacts induce cyclic elastic strains that lead to initiation of a matrix of fatigue cracks, which grows to failure by the surface fatigue phenomenon described earlier, the process is called *impact wear*. If fretting action, as described in the next paragraph, is induced by the small lateral relative displacements between two surfaces as they impact together, where the small displacements are caused by Poisson strains or small tangential "glancing" velocity components, the phenomenon is called *impact fretting*. *Impact fatigue* failure occurs when impact loading is applied repetitively to a machine member until failure occurs by the nucleation and propagation of a fatigue crack.

Fretting action may occur at the interface between any two solid bodies whenever they are pressed together by a normal force and subjected to small-amplitude cyclic relative motion with respect to each other. Fretting usually takes place in joints that are not intended to move but, because of vibrational loads or deformations, experience minute cyclic relative motions. Typically, debris produced by fretting action is trapped between the surfaces because of the small motions involved. *Fretting fatigue* failure is the premature fatigue fracture of a machine member subjected to fluctuating loads or strains together with conditions that simultaneously produce fretting action. The surface discontinuities and microcracks generated by the fretting action act as fatigue crack nuclei that propagate to failure under conditions of fatigue loading that would otherwise be acceptable. Fretting fatigue failure is an insidious failure mode because the fretting action is usually hidden within a joint where it cannot be seen and leads to premature, or even unexpected, fatigue failure of a sudden and catastrophic nature. *Fretting wear* failure results when the changes in dimensions of the mating parts, because of the presence of fretting action, become large enough to interfere with proper design function or large enough to produce geometrical stress concentration of such magnitude that failure ensues as a result of excessive local stress levels. *Fretting corrosion* failure occurs when a machine part is rendered incapable of performing its intended function because of the surface degradation of the material from which the part is made, as a result of fretting action.

Creep failure results whenever the plastic deformation in a machine member accrues over a period of time under the influence of stress and temperature until the accumulated dimensional changes interfere with the ability of the machine part to satisfactorily perform its intended function. Three stages of creep are often observed: (1) transient or primary creep during which time the rate of strain decreases, (2) steady-state or secondary creep during which time the rate of strain is virtually constant, and (3) tertiary creep during which time the creep strain rate increases, often rapidly, until rupture occurs. This terminal rupture is often called creep rupture and may or may not occur, depending on the stress–time–temperature conditions.

Thermal relaxation failure occurs when the dimensional changes due to the creep process result in the relaxation of a prestrained or prestressed member until it no longer is able to perform its intended function. For example, if the prestressed flange bolts of a high-temperature pressure vessel relax over a period of time because of creep in the bolts, so that, finally, the peak pressure surges exceed the bolt preload to violate the flange seal, the bolts will have failed because of thermal relaxation.

Stress rupture failure is intimately related to the creep process except that the combination of stress, time, and temperature is such that rupture into two parts is ensured. In stress rupture failures the combination of stress and temperature is often such that the period of steady-state creep is short or nonexistent.

Thermal shock failure occurs when the thermal gradients generated in a machine part are so pronounced that differential thermal strains exceed the ability of the material to sustain them without yielding or fracture.

Galling failure occurs when two sliding surfaces are subjected to such a combination of loads, sliding velocities, temperatures, environments, and lubricants that massive surface destruction is caused by welding and tearing, plowing, gouging, significant plastic deformation of surface asperities, and metal transfer between the two surfaces. Galling may be thought of as a severe extension of the adhesive wear process. When such action results in significant impairment to intended surface sliding or in seizure, the joint is said to have failed by galling. *Seizure* is an extension of the galling process to such severity that the two parts are virtually welded together so that relative motion is no longer possible.

Spalling failure occurs whenever a particle is spontaneously dislodged from the surface of a machine part so as to prevent the proper function of the member. Armor plate fails by spalling, for example, when a striking missile on the exposed side of an armor shield generates a stress wave that propagates across the plate in such a way as to dislodge or spall a secondary missile of lethal potential on the protected side. Another example of spalling failure is manifested in rolling contact bearings and gear teeth because of the action of surface fatigue as described earlier.

Radiation damage failure occurs when the changes in material properties induced by exposure to a nuclear radiation field are of such a type and magnitude that the machine part is no longer able to perform its intended function, usually as a result of the triggering of some other failure mode, and often related to loss in ductility associated with radiation exposure. Elastomers and polymers are typically more susceptible to radiation damage than are metals, whose strength properties are sometimes enhanced rather than damaged by exposure to a radiation field, although ductility is usually decreased.

Buckling failure occurs when, because of a critical combination of magnitude and/or point of load application, together with the geometrical configuration of a machine member, the deflection of the member suddenly increases greatly with only a slight change in load. This nonlinear response results in a buckling failure if the buckled member is no longer capable of performing its design function.

Creep buckling failure occurs when, after a period of time, the creep process results in an unstable combination of the loading and geometry of a machine part so that the critical buckling limit is exceeded and failure ensues.

Stress corrosion failure occurs when the applied stresses on a machine part in a corrosive environment generate a field of localized surface cracks, usually along grain boundaries, that render the part incapable of performing its function, often because of triggering some other failure mode. Stress corrosion is a very important type of corrosion failure mode because so many different metals are susceptible to it. For example, a variety of iron, steel, stainless-steel, copper, and aluminum alloys are subject to stress corrosion cracking if placed in certain adverse corrosive media.

Corrosion wear failure is a combination failure mode in which corrosion and wear combine their deleterious effects to incapacitate a machine part. The corrosion process often produces a hard, abrasive corrosion product that accelerates the wear, while the wear process constantly removes the protective corrosion layer from the surface, baring fresh metal to the corrosive medium and thus accelerating the corrosion. The two modes combine to make the result more serious than either of the modes would have been otherwise.

Corrosion fatigue is a combination failure mode in which corrosion and fatigue combine their deleterious effects to cause failure of a machine part. The corrosion process often forms pits and surface discontinuities that act as stress raisers that in turn accelerate fatigue failure. Furthermore, cracks in the usually brittle corrosion layer also act as fatigue crack nuclei that propagate into the base material. On the other hand, the cyclic loads or strains cause cracking and flaking of the corrosion layer, which bares fresh metal to the corrosive medium. Thus, each process accelerates the other, often making the result disproportionately serious.

Combined creep and fatigue failure is a combination failure mode in which all of the conditions for both creep failure and fatigue exist simultaneously, each process influencing the other to produce failure. The interaction of creep and fatigue is probably synergistic but is not well understood.

3 ELASTIC DEFORMATION AND YIELDING

Small changes in the interatomic spacing of a material, induced by applied forces or changing temperatures, are manifested macroscopically as elastic strain. Although the maximum elastic strain in crystalline solids, including engineering metals, is typically very small, the force required to produce the small strain is usually large; hence, the accompanying stress is large. For uniaxial loading of a machine or structural element, the total elastic deformation of the member may be found by integrating the elastic strain over the length of the element. Thus, for a uniform bar subjected to uniaxial loading the total deformation of the bar in the axial direction is

$$\Delta l = l\varepsilon \tag{1}$$

where Δl is total axial deformation of the bar, l is the original bar length, and ε is the axial elastic strain. If Δl exceeds the design-allowable axial deformation, failure will occur. For example, if the axial deformation of an aircraft gas-turbine blade, due to the centrifugal force field, exceeds the tip clearance gap, failure will occur because of force-induced elastic deformation. Likewise, if thermal expansion of the blade produces a blade-axial deformation that exceeds the tip clearance gap, failure will occur because of temperature-induced elastic deformation.

When the state of stress is more complicated, it becomes necessary to calculate the elastic strains induced by the multiaxial states of stress in three mutually perpendicular directions through the use of the generalized Hooke's law equations for isotropic materials:

$$\begin{aligned} \varepsilon_x &= \frac{1}{E}[\sigma_x - \nu(\sigma_y + \sigma_z)] \\ \varepsilon_y &= \frac{1}{E}[\sigma_y - \nu(\sigma_x + \sigma_z)] \\ \varepsilon_z &= \frac{1}{E}[\sigma_z - \nu(\sigma_x + \sigma_y)] \end{aligned} \tag{2}$$

where σ_x, σ_y, and σ_z are the normal stresses in the three coordinate directions, E and ν are Young's modulus and Poisson's ratio, respectively, and ε_x, ε_y, and ε_z are the elastic strains in the three coordinate directions. Again, total elastic deformation of a member in any of the coordinate directions may be found by integrating the strain over the member's length in that direction. If the change in length of the member in any direction exceeds the design-allowable deformation in that direction, failure will occur. The use of commercial finite-element analysis software packages is one commonly used means of determining both the elastic strains produced in a structural element and the subsequent elastic deformations produced.

If applied loads reach certain critical levels, the atoms within the microstructure may be moved into new equilibrium positions and the induced strains are not fully recovered upon release of the loads. Such permanent strains, usually the result of slip, are called plastic strains, and the macroscopic permanent deformation due to plastic strain is called yielding. If applied loads are increased even more, the plastic deformation process may be carried to the point of instability where *necking* begins: Internal voids form and slowly coalesce to finally produce a ductile rupture of the loaded member.

After plastic deformation has been initiated, Eqs. (2) are no longer valid and the predictions of plastic strains and deformations under multiaxial states of stress are more difficult. If a designer can tolerate a prescribed plastic deformation without experiencing failure, these plastic deformations may be determined using plasticity theory. Many commercial finite-element analysis software packages now posses the capability to compute both plastic strains and deformations for a prescribed nonlinear elastic-plastic constitutive relation.

For the case of simple uniaxial loading, the onset of yielding may be accurately predicted to occur when the uniaxial normal stress reaches a value equal to the yield strength of the material read from an engineering stress–strain curve. If the loading is more complicated, and a multiaxial state of stress is produced by the loads, the onset of yielding may no longer be predicted by comparing any one of the normal stress components with uniaxial material yield strength, not even the maximum principal normal stress. Onset of yielding for multiaxially stressed critical points in a machine or structure is more accurately predicted through the use of a *combined stress theory of failure,* which has experimentally been validated for the prediction of yielding. The two most widely accepted theories for predicting the onset of yielding are the distortion energy theory (also called the octahedral shear stress theory or the von Mises criterion) and the maximum shearing stress theory (also called the Tresca criterion). The distortion energy theory is somewhat more accurate while the maximum shearing stress theory may be slightly easier to use and is more conservative .

In words, the distortion energy theory may be expressed as follows: *Failure is predicted to occur in the multiaxial state of stress when the distortion energy per unit volume becomes equal to or exceeds the distortion energy per unit volume at the time of failure in a simple uniaxial stress test using a specimen of the same material.*

Mathematically, the distortion energy theory may be formulated as: *Failure is predicted by the distortion energy theory to occur if*

$$\lfloor(\sigma_1 - \sigma_2)^2 + (\sigma_1 - \sigma_3)^2 + (\sigma_3 - \sigma_1)^2\rfloor \geq 2\sigma_f^2 \tag{3}$$

The maximum shearing stress theory may be stated in words as: *Failure is predicted to occur in the multiaxial state of stress when the maximum shearing stress magnitude becomes equal to or exceeds the maximum shearing stress magnitude at the time of failure in a simple uniaxial stress test using a specimen of the same material.*

Mathematically, the maximum shearing stress theory becomes: *Failure is predicted by the maximum shearing stress theory to occur if*

$$\sigma_1 - \sigma_3 \geq \sigma_f \tag{4}$$

where σ_1, σ_2, and σ_3 are the principal stresses at a point, ordered such that $\sigma_1 \geq \sigma_2 \geq \sigma_3$, σ_f is the uniaxial failure strength in tension.

Comparisons of these two failure theories with experimental data on yielding are shown in Fig. 1 for a variety of materials and different biaxial states of stress.

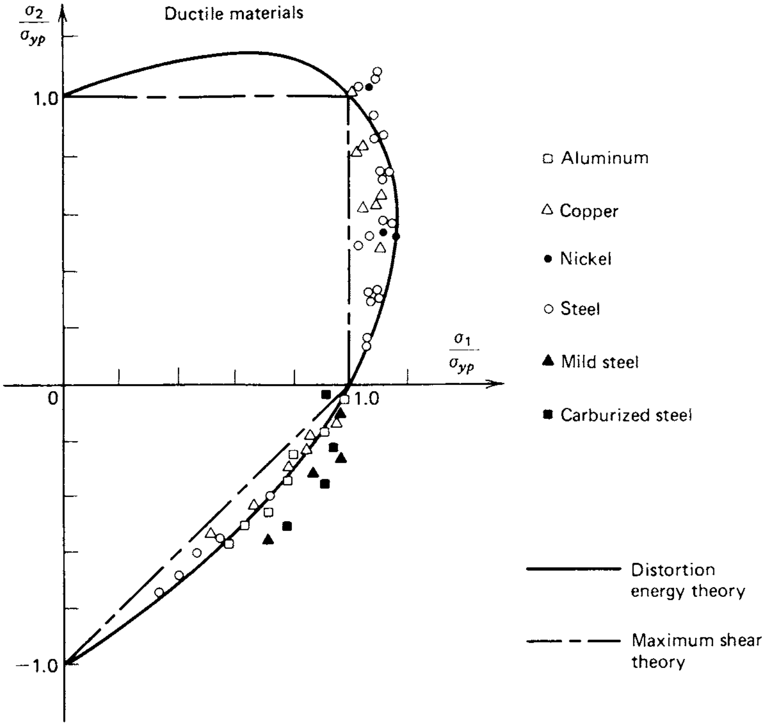


Fig. 1 Comparison of biaxial yield strength data with theories of failure for a variety of ductile materials.

4 FRACTURE MECHANICS AND UNSTABLE CRACK GROWTH

When the material behavior is brittle rather than ductile, the mechanics of the failure process are much different. Instead of the slow coalescence of voids associated with ductile rupture, brittle fracture proceeds by the high-velocity propagation of a crack across the loaded member. If the material behavior is clearly brittle, fracture may be predicted with reasonable accuracy through use of the maximum normal stress theory of failure. In words, the maximum normal stress theory may be expressed as follows: *Failure is predicted to occur in the multiaxial state of stress when the maximum principal normal stress becomes equal to or exceeds the maximum normal stress at the time of failure in a simple uniaxial stress test using a specimen of the same material.*

Mathematically, the maximum normal stress theory becomes: *Failure is predicted by the maximum normal stress theory to occur if*

$$\sigma_1 \geq \sigma_t \qquad |\sigma_3| \geq \sigma_c \tag{5}$$

where σ_1, σ_2, and σ_3 are the principal stresses at a point, ordered such that $\sigma_1 \geq \sigma_2 \geq \sigma_3$, σ_t is the uniaxial failure strength in tension, and σ_c is the uniaxial failure strength in compression. Comparison of this failure theory with experimental data on brittle fracture for different biaxial states of stress is shown in Fig. 2.

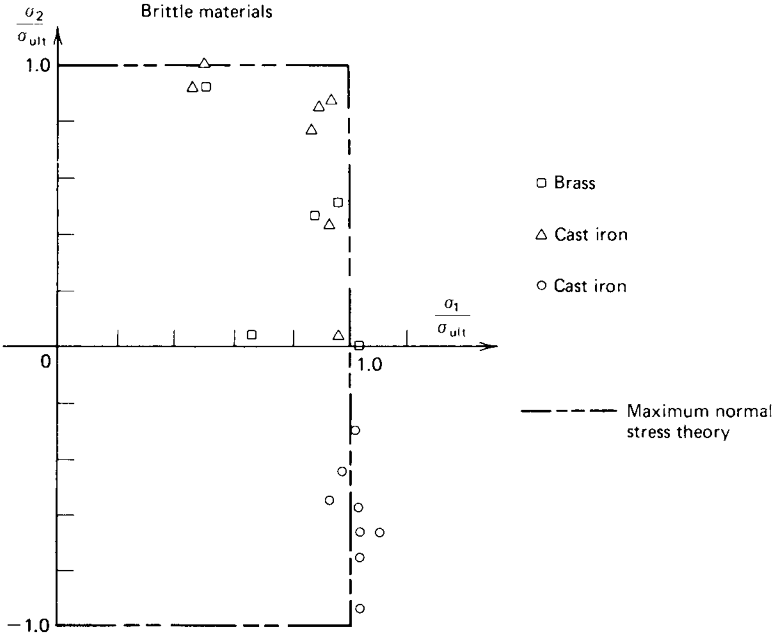


Fig. 2 Comparison of biaxial brittle fracture strength data with maximum normal stress theory for several brittle materials.

On the other hand, more recent experience has led to the understanding that nominally ductile materials may also fail by a brittle fracture response in the presence of cracks or flaws if the combination of crack size, geometry of the part, temperature, and/or loading rate lies within certain critical regions. Furthermore, the development of higher-strength structural alloys, the wider use of welding, and the use of thicker sections in some cases have combined their influence to reduce toward a critical level the capacity of some structural members to accommodate local plastic strain without fracture. At the same time, fabrication by welding, residual stresses due to machining, and assembly mismatch in production have increased the need for accommodating local plastic strain to prevent failure. Fluctuating service loads of greater severity and more aggressive environments have also contributed to unexpected fractures.

An important observation in studying fracture behavior is that the magnitude of the nominal applied stress that causes fracture is related to the size of the crack or cracklike flaw within the structure.[2–4] For example, observations of the behavior of central through-the-thickness cracks, oriented normal to the applied tensile stress in steel and aluminum plates, yielded the results shown in Figs. 3 and 4. In these tests, as the tensile loading on the precracked plates was slowly increased, the crack initially extended slowly for a time and then abruptly extended to failure by rapid crack propagation. The slow stable crack growth or tearing was characterized by speeds of the order of fractions of an inch per minute. The rapid crack propagation was characterized by speeds of the order of hundreds of feet per second. The data of Figs. 3 and 4 indicate that for longer initial crack lengths the fracture stress (the stress corresponding to the onset of stable tearing) was lower. For the aluminum alloy the fracture stress was less

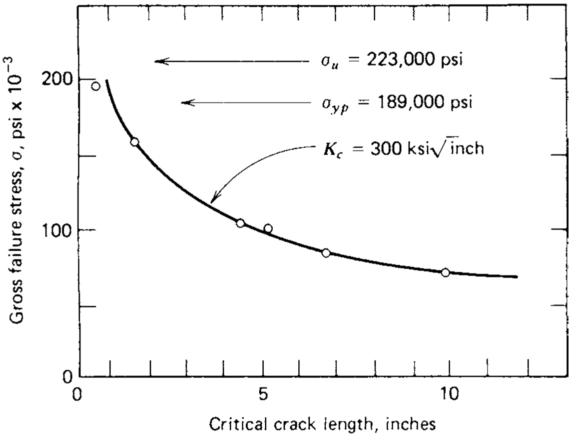


Fig. 3 Influence of crack length on gross failure stress for center-cracked steel plate. (After Ref. 5, copyright ASTM, adapted with permission)

than the yield strength for cracks longer than about 0.75 in. For the steel alloy the fracture stress was less than the yield strength for cracks longer than about 0.5 in. In both cases, for shorter cracks the fracture stress approaches the ultimate strength of the material determined from a conventional uniaxial tension test.

Experience has shown that the onset of stable tearing establishes an important material property termed *fracture toughness*. The fracture toughness may be used as a design criterion in fracture prevention, just as the yield strength is used as a design criterion in prevention of yielding of a ductile material under static loading. It should be noted that not all materials and or specimen types exhibit stable tearing. Many materials and or specimen configurations exhibit rapid crack propagation without any evidence of prior stable tearing.

In many cases slow crack propagation occurs by means other than stable tearing, especially under conditions of fluctuating loads and/or aggressive environments. In analyses and predictions involving fatigue failure phenomena,

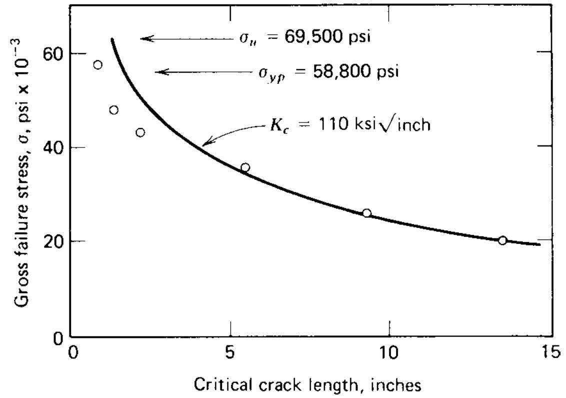


Fig. 4 Influence of crack length on gross failure stress for center-cracked aluminum plate. (After Ref. 5, copyright ASTM, adapted with permission)

characterization of the rate of slow crack extension and the initial flaw size, together with critical crack size, are used to determine the useful life of a component or structure subjected to fluctuating loads. The topic of fatigue crack propagation is discussed further in Section 5.

The simplest useful model for the stresses near the tip of a crack is based on the assumptions of linear elastic material behavior and a two-dimensional analysis; thus, the procedure is often referred to as linear elastic fracture mechanics. Although the validity of the linear elastic assumption may be questioned in view of plastic zone formation at the tip of a crack in any real engineering material, as long as *small-scale yielding* occurs, that is, as long as the plastic zone size remains small compared to the dimensions of the crack, the linear elastic model gives good engineering results. Thus, the small-scale yielding concept implies that the small plastic zone is confined within a linear elastic field surrounding the crack tip. If the material properties, section size, loading conditions, and environment combine in such a way that large-scale plastic zones are formed, the basic assumptions of linear elastic fracture mechanics are violated, and elastic-plastic fracture mechanics methods must be employed.[4]

Three basic types of stress fields can be defined for crack-tip stress analysis, each one associated with a distinct mode of crack deformation, as illustrated in Fig. 5. The opening mode, mode I, is associated with local displacement in which the crack surfaces move directly apart, as shown in Fig. 5*a*. The sliding mode, mode II, is developed when crack surfaces slide over each other in a direction perpendicular to the leading edge of the crack, as shown in Fig. 5*b*. The tearing mode, mode III, is characterized by crack surfaces sliding with respect to each other in a direction parallel to the leading edge of the crack, as shown in Fig. 5*c*. Superposition of these three modes will fully describe the most general three-dimensional case of local crack-tip deformation and stress field, although mode I is most common.

In terms of the coordinates shown in Fig. 6, the stresses near the crack tip for mode I loading may be written as[2]

$$\sigma_x = \frac{K_\mathrm{I}}{\sqrt{2\pi r}} \cos\frac{\theta}{2}\left[1 - \sin\frac{\theta}{2}\sin\frac{3\theta}{2}\right] \tag{6}$$

$$\sigma_y = \frac{K_\mathrm{I}}{\sqrt{2\pi r}} \cos\frac{\theta}{2}\left[1 + \sin\frac{\theta}{2}\sin\frac{3\theta}{2}\right] \tag{7}$$

$$\tau_{xy} = \frac{K_\mathrm{I}}{\sqrt{2\pi r}} \sin\frac{\theta}{2}\cos\frac{\theta}{2}\cos\frac{3\theta}{2} \tag{8}$$

The parameter K_I is known as the mode I stress intensity factor. This parameter represents the strength of the stress field surrounding the tip of the crack. Since fracture is induced by the crack-tip stress field, the stress intensity factor is the primary correlation parameter used in current practice.

In general, the expressions for the stress intensity factor are of the form

$$K_\mathrm{I} = C\sigma\sqrt{\pi a} \tag{9}$$

where a is the crack size, σ is the gross-section stress, and C is dependent on

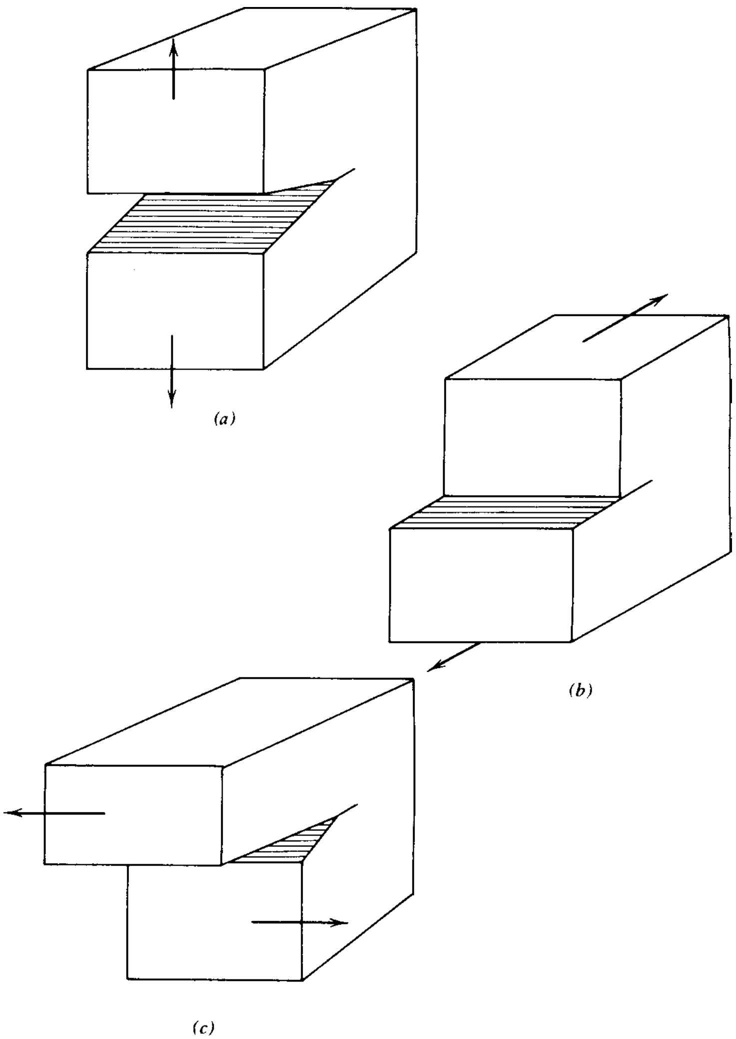


Fig. 5 Basic modes of crack displacement: (*a*) mode I, (*b*) mode II, and (*c*) mode III.

the type of loading and the geometry away from the crack. Much work has been completed in determining values of *C* for a wide variety of conditions. (See, for example, Ref. 5.)

Many commercial finite-element analysis software packages possess special crack-tip elements allowing the numerical computation of stress intensity factors. A discussion of some of the techniques employed within these software packages is given by Anderson.[4] Through the use of weight functions,[6,35] stress intensity factors may also be computed easily using numerical integration and the stresses that would exist in the uncracked body.

From Eq. (9), the stress intensity factor increases proportionally with gross nominal stress σ and also is a function of the crack length a. The value of K_I associated with the onset of fracture has been designated the *critical stress intensity*, K_C. As noted earlier in Figs. 3 and 4, the fracture of specimens with

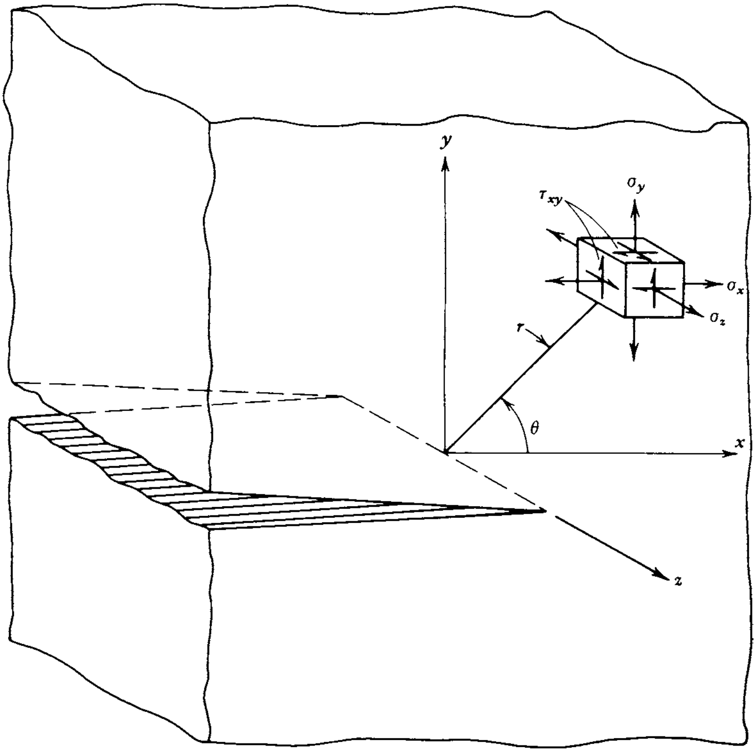


Fig. 6 Coordinates measured from leading edge of a crack.

different crack lengths occurs at different values of gross-section stress, but at a constant value of K_C. Thus, K_C provides a single-parameter fracture criterion that allows the prediction of fracture based on (9). That is, *fracture is predicted to occur if*

$$K_I \geq K_C \tag{10}$$

In studying material behavior, one finds that for a given material, as the specimen thickness is increased, the critical stress intensity K_C decreases to a lower limiting value. This lower limiting value defines a basic material property K_{IC}, the *plane-strain fracture toughness* for the material. Standard test methods have been established for the determination of K_{IC} values.[7] A few data are shown in Table 1. Useful compilations of fracture toughness values have been prepared by several organizations and individuals. These include Refs. 9–15.

For the plane-strain fracture toughness K_{IC} to be a valid failure prediction criterion for a specimen or a machine part, plane-strain conditions must exist at the crack tip; that is, the material must be thick enough to ensure plane-strain conditions. It has been estimated [7] empirically that for plane-strain conditions the minimum material thickness B must be

Table 1 Yield Strength and Plane-Strain Fracture Toughness Data for Selected Engineering Alloys[8,9]

Alloy	Form	Test Temperature		σ_{yp}		K_{IC}	
		°F	°C	ksi	MPa	ksi$\sqrt{\text{in}}$	MPa$\sqrt{\text{m}}$
4340 (500°F temper) steel	Plate	70	21	217–238	1495–1640	45–57	50–63
4340 (800°F temper) steel	Forged	70	21	197–211	1360–1455	72–83	79–91
D6AC (1000°F temper) steel	Plate	170	21	217	1495	93	102
D6AC (1000°F temper) steel	Plate	−65	−54	228	1570	56	62
A 538 steel				250	1722	100	111
2014-T6 aluminum	Forged	75	24	64	440	28	31
2024-T351 aluminum	Plate	80	27	54–56	370–385	28–40	31–44
7075-T6 aluminum				85	585	30	33
7075-T651 aluminum	Plate	70	21	75–81	515–560	25–28	27–31
7075-T7351 aluminum	Plate	70	21	58–66	300–455	28–32	31–35
Ti-6Al-4V titanium	Plate	74	23	119	820	96	106

$$B \geq 2.5 \left(\frac{K_{\mathrm{IC}}}{\sigma_{\mathrm{yp}}}\right)^2 \tag{11}$$

where σ_{yp} is the material yield strength.

If the material is not thick enough to meet the criterion of (11), plane stress is a more likely state of stress at the crack tip; and K_{C}, the critical stress intensity factor for failure prediction under plane stress conditions, may be estimated using a semiempirical relationship[16]:

$$K_{\mathrm{C}} = K_{\mathrm{IC}} \left[1 + \frac{1.4}{B^2}\left(\frac{K_{\mathrm{IC}}}{\sigma_{\mathrm{yp}}}\right)^4\right]^{1/2} \tag{12}$$

As long as the crack-tip plastic zone remains in the regime of small-scale yielding, this estimation procedure provides a good design approach. For conditions that result in large crack-tip plastic zones (large applied stresses, large crack lengths), performing a failure assessment using linear elastic fracture mechanics (LEFM) is invalid and potentially nonconservative. A general rule of thumb is that plasticity effects become significant when the applied stresses approach 50% of the yield stress, but this is by no means a universal rule.[4] When small-scale yielding is not generated at the crack tip, a better design approach would involve the implementation of an appropriate elastic-plastic fracture mechanics (EPFM) procedure.

5 FATIGUE

Static or quasi-static loading is rarely observed in modern engineering practice, making it essential for the designer to address himself or herself to the implications of repeated loads, fluctuating loads, and rapidly applied loads. By far, the majority of engineering design projects involve machine parts subjected to fluctuating or cyclic loads. Such loading induces fluctuating or cyclic stresses that often result in failure by fatigue.

Fatigue failure investigations over the years have led to the observation that the fatigue process actually embraces two domains of cyclic stressing or straining that are significantly different in character, and in each of which failure is probably produced by different physical mechanisms. One domain of cyclic loading is that for which significant plastic strain occurs during each cycle. This domain is associated with high loads and short lives, or low numbers of cycles to produce fatigue failure, and is commonly referred to as *low-cycle fatigue*. The other domain of cyclic loading is that for which the strain cycles are largely confined to the elastic range. This domain is associated with lower loads and long lives, or high numbers of cycles to produce fatigue failure, and is commonly referred to as *high-cycle fatigue*. Low-cycle fatigue is typically associated with cycle lives from 1 up to about 10^4 cycles. Fatigue may be characterized as a progressive failure phenomenon that proceeds by the *initiation* and *propagation* of cracks to an unstable size. Although there is not complete agreement on the microscopic details of the initiation and propagation of the cracks, processes of reversed slip and dislocation interaction appear to produce fatigue nuclei from which cracks may grow. Finally, the crack length reaches a critical dimension and one additional cycle then causes complete failure. The final failure region will typically show evidence of plastic deformation produced just prior to final separation. For ductile materials the final fracture area often appears as a shear lip produced by crack propagation along the planes of maximum shear.

Although designers find these basic observations of great interest, they must be even more interested in the macroscopic phenomenological aspects of fatigue failure and in avoiding fatigue failure during the design life. Some of the macroscopic effects and basic data requiring consideration in designing under fatigue loading include:

1. The effects of a simple, completely reversed alternating stress on the strength and properties of engineering materials
2. The effects of a steady stress with a superposed alternating component, that is, the effects of cyclic stresses with a nonzero mean
3. The effects of alternating stresses in a multiaxial state of stress
4. The effects of stress gradients and residual stresses, such as imposed by shot peening or cold rolling
5. The effects of stress raisers, such as notches, fillets, holes, threads, riveted joints, and welds.
6. The effects of surface finish, including the effects of machining, cladding, electroplating, and coating
7. The effects of temperature on fatigue behavior of engineering materials
8. The effects of size of the structural element
9. The effects of accumulating cycles at various stress levels and the permanence of the effect
10. The extent of the variation in fatigue properties to be expected for a given material
11. The effects of humidity, corrosive media, and other environmental factors
12. The effects of interaction between fatigue and other modes of failure, such as creep, corrosion, and fretting

5.1 Fatigue Loading and Laboratory Testing

Faced with the design of a fatigue-sensitive element in a machine or structure, a designer is very interested in the fatigue response of engineering materials to various loadings that might occur throughout the design life of the machine under consideration. That is, the designer is interested in the effects of various *loading spectra* and associated *stress spectra,* which will in general be a function of the design configuration and the operational use of the machine.

Perhaps the simplest fatigue stress spectrum to which an element may be subjected is a zero-mean sinusoidal stress–time pattern of constant amplitude and fixed frequency, applied for a specified number of cycles. Such a stress–time pattern, often referred to as a completely reversed cyclic stress, is illustrated in Fig. 7*a*. Utilizing the sketch of Fig. 7*b*, we can conveniently define several useful terms and symbols; these include:

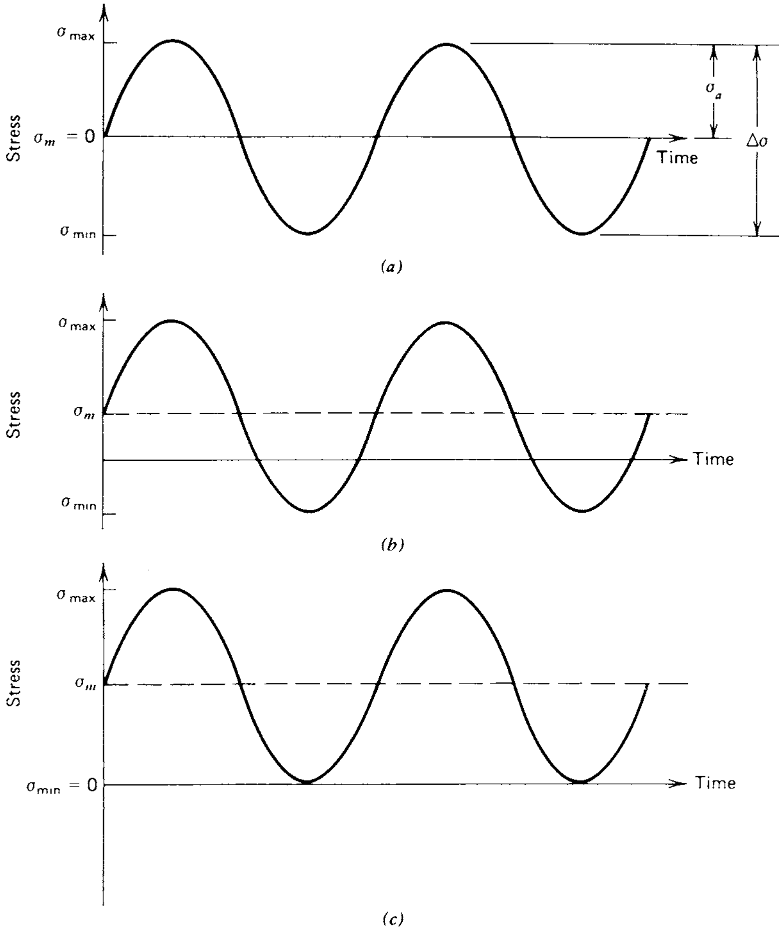


Fig. 7 Several constant-amplitude stress–time patterns of interest: (*a*) completely reversed, $R = -1$, (*b*) nonzero mean stress, (*c*) released tension, $R = 0$.

$\sigma_{\max}$ = maximum stress in the cycle

σ_m = mean stress = $(\sigma_{\max} + \sigma_{\min})/2$

$\sigma_{\min}$ = minimum stress in the cycle

σ_a = alternating stress amplitude = $(\sigma_{\max} - \sigma_{\min})/2$

$\Delta\sigma$ = range of stress = $\sigma_{\max} - \sigma_{\min}$

R = stress ratio = $\sigma_{\min}/\sigma_{\max}$

Any two of the quantities just defined, except the combinations σ_a and $\Delta\sigma$, are sufficient to describe completely the stress–time pattern.

More complicated stress–time patterns are produced when the mean stress, the stress amplitude, or both change during the operational cycle, as illustrated in Fig. 8. It may be noted that this stress–time spectrum is beginning to approach

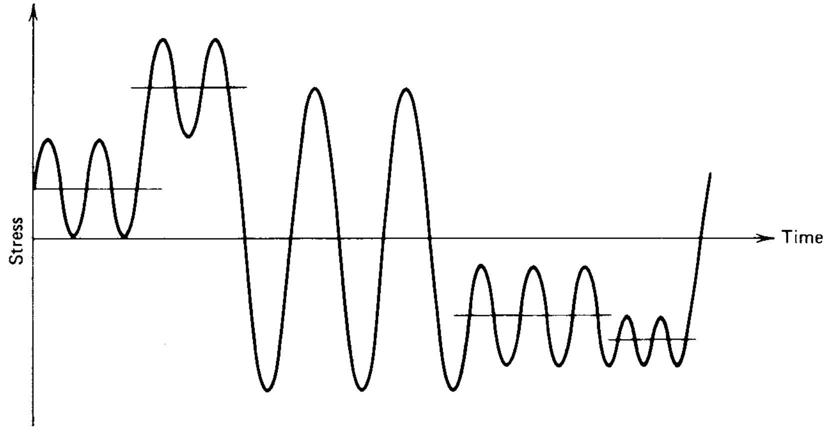


Fig. 8 Stress–time pattern in which both mean and amplitude change to produce a more complicated stress spectrum.

a degree of realism. Finally, in Fig. 9 a sketch of a realistic stress spectrum is given. This type of quasi-random stress–time pattern might be encountered in an airframe structural member during a typical mission including refueling, taxi, takeoff, gusts, maneuvers, and landing. The obtaining of useful, realistic data is a challenging task in itself. Instrumentation of existing machines, such as operational aircraft, provide some useful information to the designer if his or her mission is similar to the one performed by the instrumented machine. Recorded

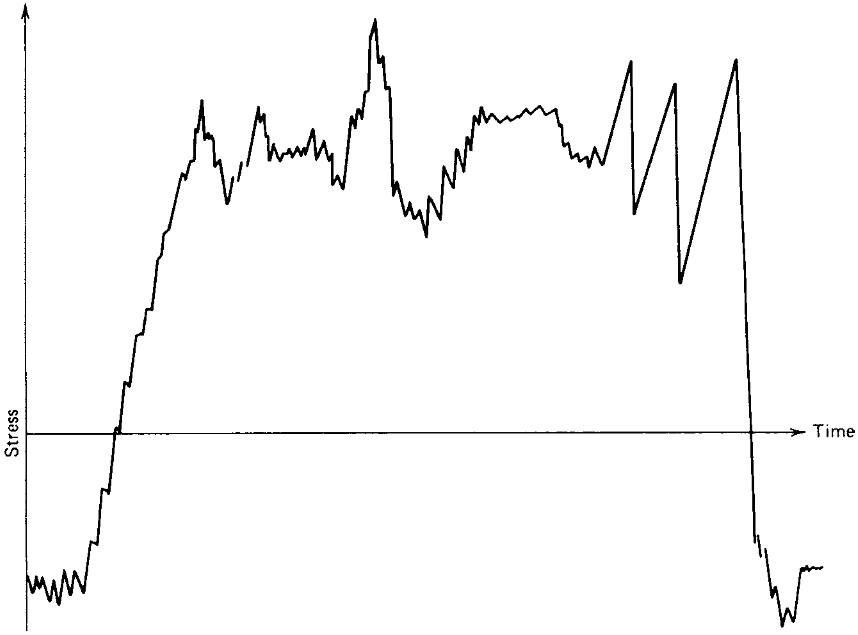


Fig. 9 A quasi-random stress–time pattern typical of an aircraft during a given mission.

data from accelerometers, strain gauges, and other transducers may in any event provide a basis from which a statistical representation can be developed and extrapolated to future needs if the fatigue processes are understood.

Basic data for evaluating the response of materials, parts, or structures are obtained from carefully controlled laboratory tests. Various types of testing machines and systems commonly used include:

1. Rotating-bending machines
    - **a.** Constant bending moment type
    - **b.** Cantilever bending type
2. Reciprocating-bending machines
3. Axial direct-stress machines
    - **a.** Brute-force type
    - **b.** Resonant type
4. Vibrating shaker machines
    - **a.** Mechanical type
    - **b.** Electromagnetic type
5. Repeated torsion machines
6. Multiaxial stress machines
7. Computer-controlled closed-loop machines
8. Component testing machines for special applications
9. Full-scale or prototype fatigue testing systems

Computer-controlled fatigue testing machines are widely used in all modern fatigue testing laboratories. Usually such machines take the form of precisely controlled hydraulic systems with feedback to electronic controlling devices capable of producing and controlling virtually any strain–time, load–time, or displacement–time pattern desired. A schematic diagram of such a system is shown in Fig. 10.

Special testing machines for component testing and full-scale prototype testing are not found in the general fatigue testing laboratory. These systems are built up especially to suit a particular need, for example, to perform a full-scale fatigue test of a commercial jet aircraft.

It may be observed that fatigue testing machines range from very simple to very complex. The very complex testing systems, used, for example, to test a full-scale prototype, produce very specialized data applicable only to the particular prototype and test conditions used; thus, for the particular prototype and test conditions the results are very accurate, but extrapolation to other test conditions and other pieces of hardware is difficult, if not impossible. On the other hand, simple smooth-specimen laboratory fatigue data are very general and can be utilized in designing virtually any piece of hardware made of the specimen material. However, to use such data in practice requires a quantitative knowledge of many pertinent differences between the laboratory and the application, including the effects of nonzero mean stress, varying stress amplitude, environment, size, temperature, surface finish, residual stress pattern, and others. Fatigue

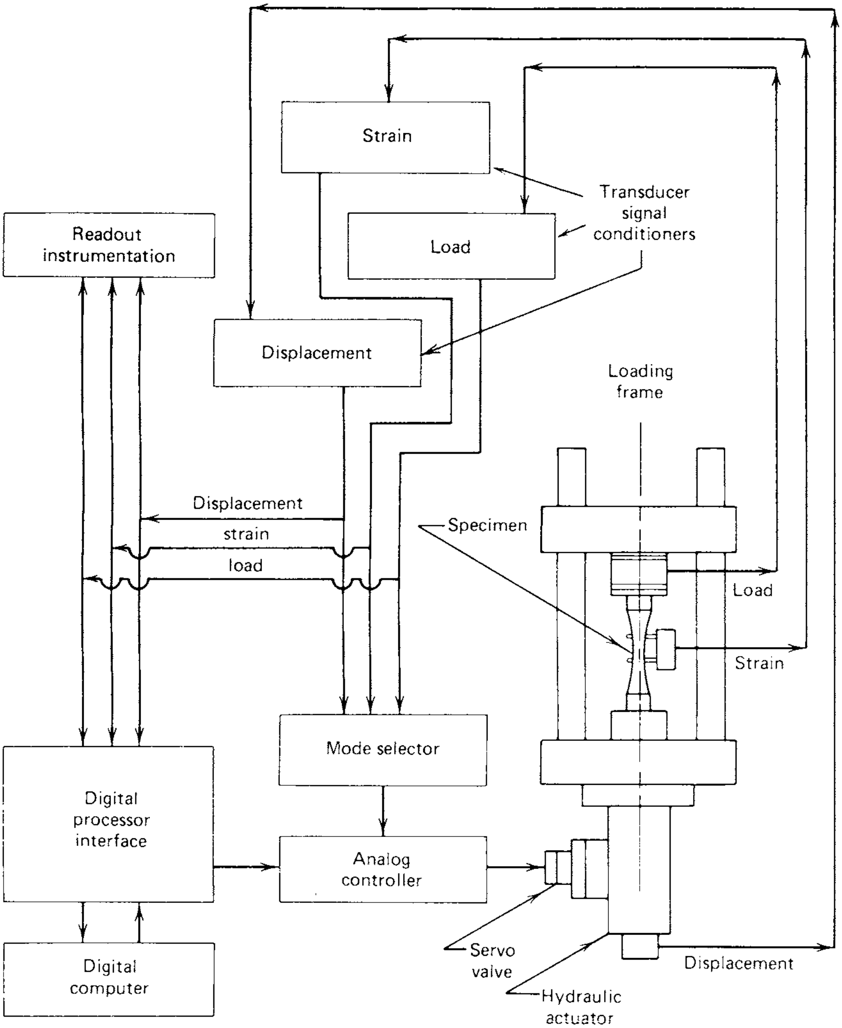


Fig. 10 Schematic diagram of a computer-controlled closed-loop fatigue testing machine.

testing is performed at the extremely simple level of smooth-specimen testing, the extremely complex level of full-scale prototype testing, and everywhere in the spectrum between. Valid arguments can be made for testing at all levels.

5.2 *S–N–P* Curves: A Basic Design Tool

Basic fatigue data in the high-cycle life range can be conveniently displayed on a plot of cyclic stress level versus the logarithm of life, or alternatively, on a log-log plot of stress versus life. These plots, called *S–N* curves, constitute design information of fundamental importance for machine parts subjected to repeated loading. Because of the scatter of fatigue life data at any given stress level, it must be recognized that there is not only one *S–N* curve for a given material, but a family of *S–N* curves with probability of failure as the parameter.

These curves are called the *S–N–P* curves, or curves of constant probability of failure on a stress-versus-life plot. A representative family of *S–N–P* curves is illustrated in Fig. 11. It should also be noted that references to the "*S–N* curve" in the literature generally refer to the mean curve unless otherwise specified. Details regarding fatigue testing and the experimental generation of *S–N–P* curves may be found in Ref. 1.

The mean *S–N* curves sketched in Fig. 12 distinguish two types of material response to cyclic loading commonly observed. The ferrous alloys and titanium exhibit a steep branch in the relatively short life range, leveling off to approach a stress asymptote at longer lives. This stress asymptote is called the *fatigue limit* or *endurance limit* and is the stress level below which an infinite number of cycles can be sustained without failure. The nonferrous alloys do not exhibit an asymptote, and the curve of stress versus life continues to drop off indefinitely. For such alloys there is no fatigue limit, and failure as a result of cyclic load is only a matter of applying enough cycles. All materials, however, exhibit a relatively flat curve in the long-life range.

To characterize the failure response of nonferrous materials, and of ferrous alloys in the finite-life range, the term *fatigue strength at a specified life,* σ_N, is used. The term fatigue strength identifies the stress level at which failure will occur at the specified life. The specification of *fatigue strength* without specifying the corresponding life is meaningless. The specification of a *fatigue limit* always implies infinite life.

5.3 Factors That Affect *S–N–P* Curves

There are many factors that may influence the fatigue failure response of machine parts or laboratory specimens, including material composition, grain size

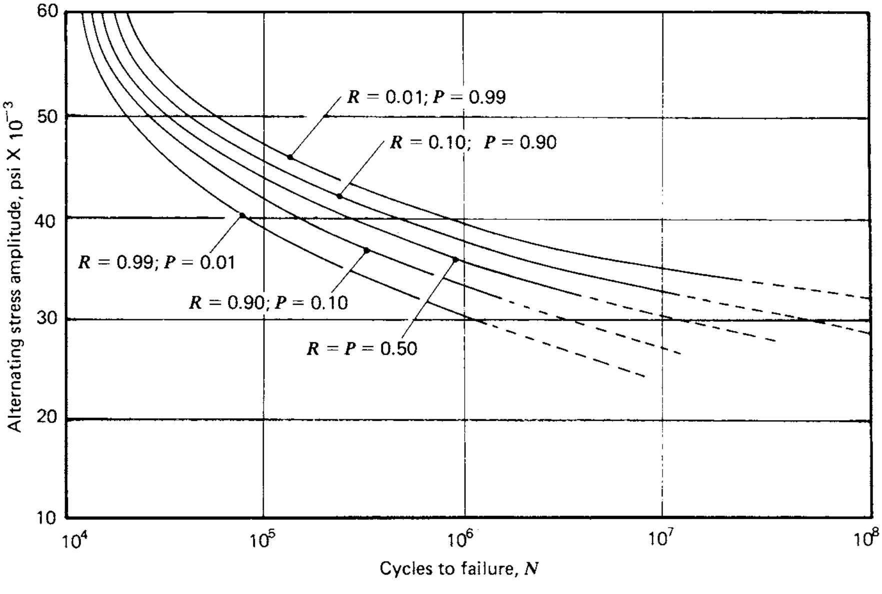


Fig. 11 Family of *S–N–P* curves or *R–S–N* curves, for 7075-T6 aluminum alloy. Note: P = probability of failure; R = reliability = $1 - P$. (Adapted from Ref. 17, with permission from John Wiley & Sons, Inc.)

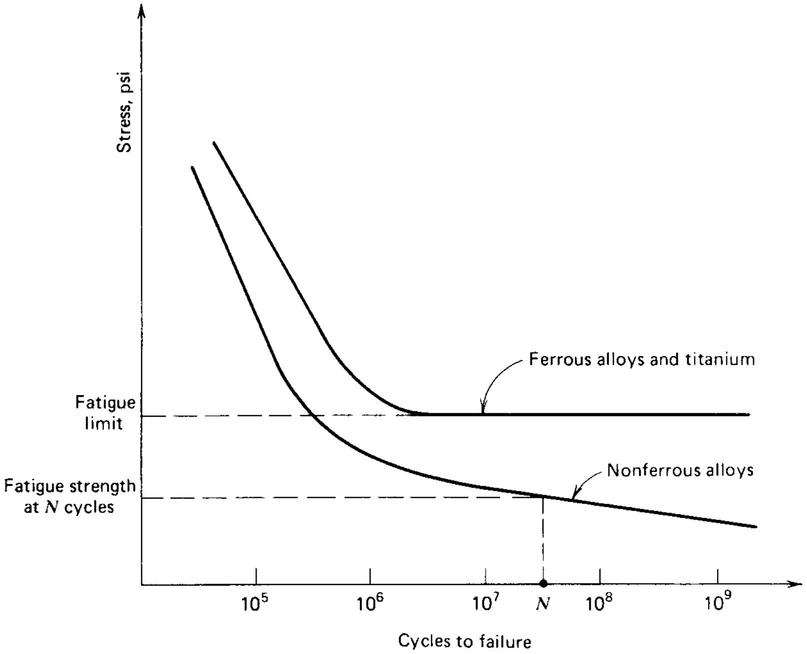


Fig. 12 Two types of material response to cyclic loading.

and grain direction, heat treatment, welding, geometrical discontinuities, size effects, surface conditions, residual surface stresses, operating temperature, corrosion, fretting, operating speed, configuration of the stress–time pattern, nonzero mean stress, and prior fatigue damage. Typical examples of how some of these factors may influence fatigue response are shown in Figs. 13 through 19. It is usually necessary to search the literature and existing databases to find the information required for a specific application, and it may be necessary to undertake experimental testing programs to produce data where they are unavailable.

5.4 Nonzero Mean Stress

Most basic fatigue data collected in the laboratory are for completely reversed alternating stresses, that is, zero mean cyclic stresses. Most service applications involve nonzero mean cyclic stresses. It is therefore very important to a designer to know the influence of mean stress on fatigue behavior so that he or she can utilize basic completely reversed laboratory data in designing machine parts subjected to nonzero mean cyclic stresses.

If a designer is fortunate enough to find test data for his or her proposed material under the mean stress conditions and design life of interest, the designer should, of course, use these data. Such data are typically presented on so-called *master diagrams* or *constant life diagrams* for the material. A master diagram for a 4340 steel alloy is shown in Fig. 20. An alternative means of presenting this type of fatigue data is illustrated in Fig. 21 for a 4130 steel alloy.

If data are not available to the designer, he or she may estimate the influence of nonzero mean stress by any one of several empirical relationships that relate

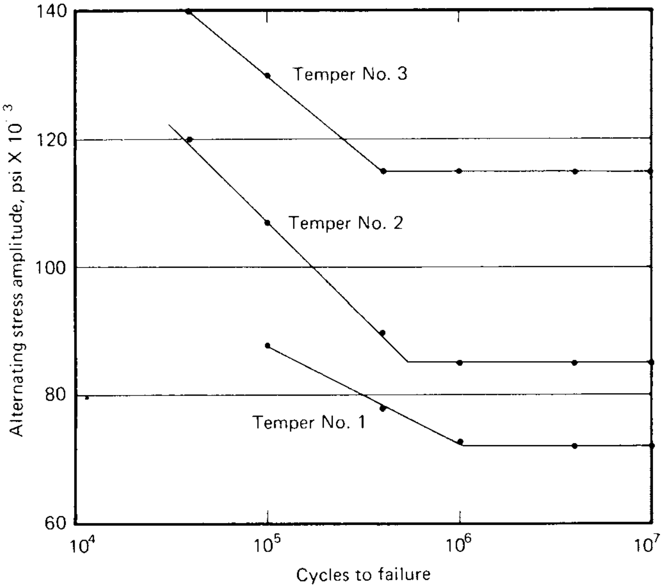


Fig. 13 Effects of heat treatment on the S–N curve of oil quenched SAE 4130 steel. Temper No. 1: S_u = 129 ksi; temper No. 2: S_u = 150 ksi; temper No. 3: S_u = 206 ksi.

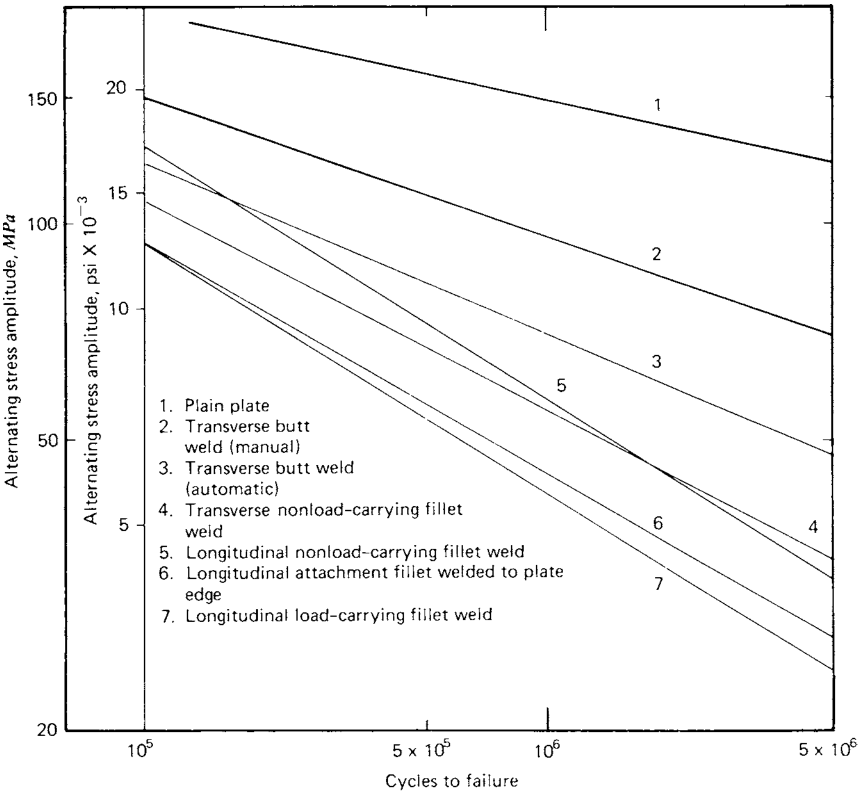


Fig. 14 Effects of welding detail on the S–N curve of structural steel, with yield strength in the range of 30–52 ksi. Tests were released tension (R = 0). (Data from Ref. 18.)

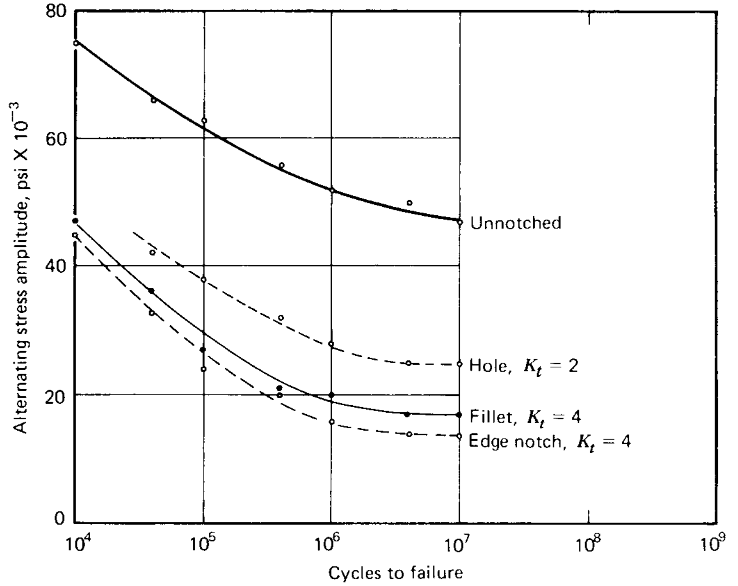


Fig. 15 Effects of geometrical discontinuities on the *S–N* curve of SAE 4130 steel sheet, tested under completely reversed axial loading. Specimen dimensions (t = thickness, w = width, r = notch radius); unnotched: t = 0.075 in., w = 1.5 in.; hole: t = 0.075 in., w = 4.5 in., r = 1.5 in.; fillet: t = 0.075 in., w_{net} = 1.5 in., w_{gross} = 2.25 in., r = 0.0195 in.; edge notch: t = 0.075 in., w_{net} = 1.5 in., w_{gross} = 2.25 in., r = 0.057 in. (Data from Ref. 19.)

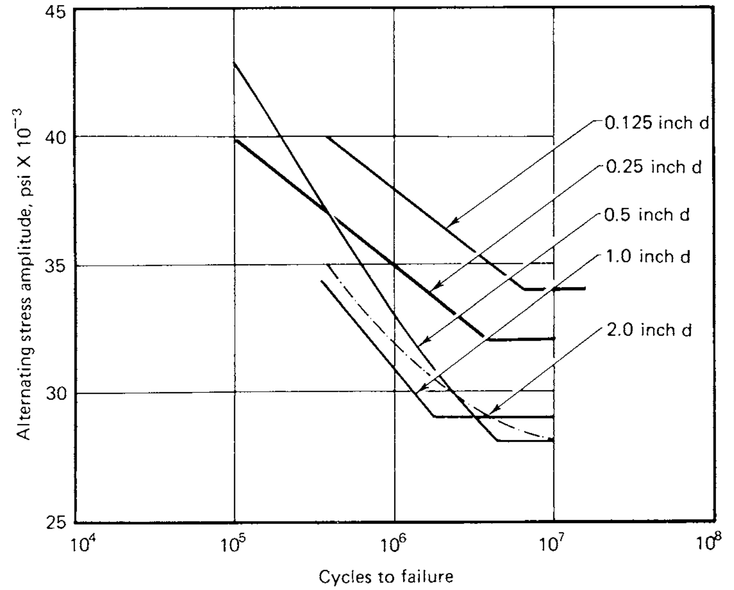


Fig. 16 Size effects on the *S–N* curve of SAE 1020 steel specimens cut from a 3.5-in. diameter hot-rolled bar, testing in rotating bending. (Data from Ref. 20.)

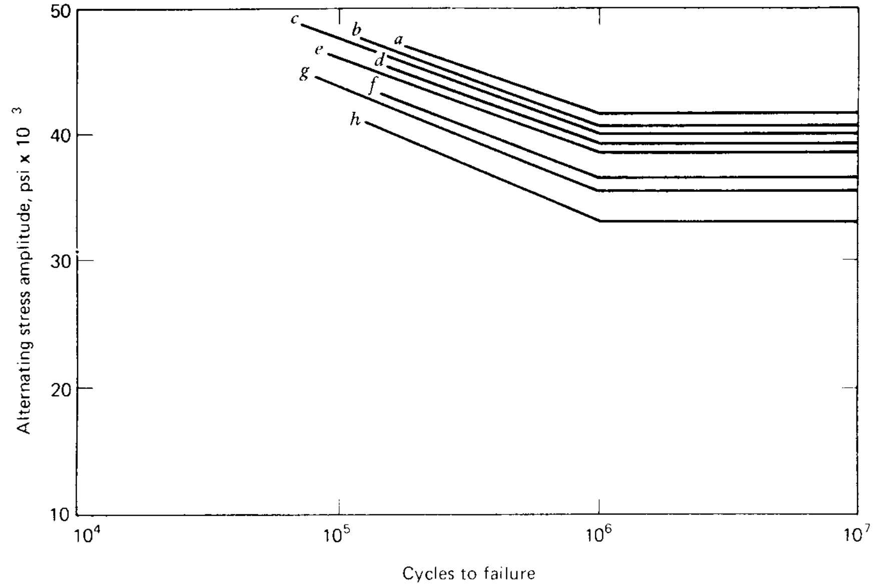


Fig. 17 Effect of surface finish on the S–N curve of carbon steel specimens, testing in rotating bending: (*a*) high polish, longitudinal direction; (*b*) FF emery finish; (*c*) No. 1 emery finish; (*d*) coarse emery finish; (*e*) smooth file; (*f*) as-turned; (*g*) bastard file; (*h*) coarse file. (Data from Ref. 21.)

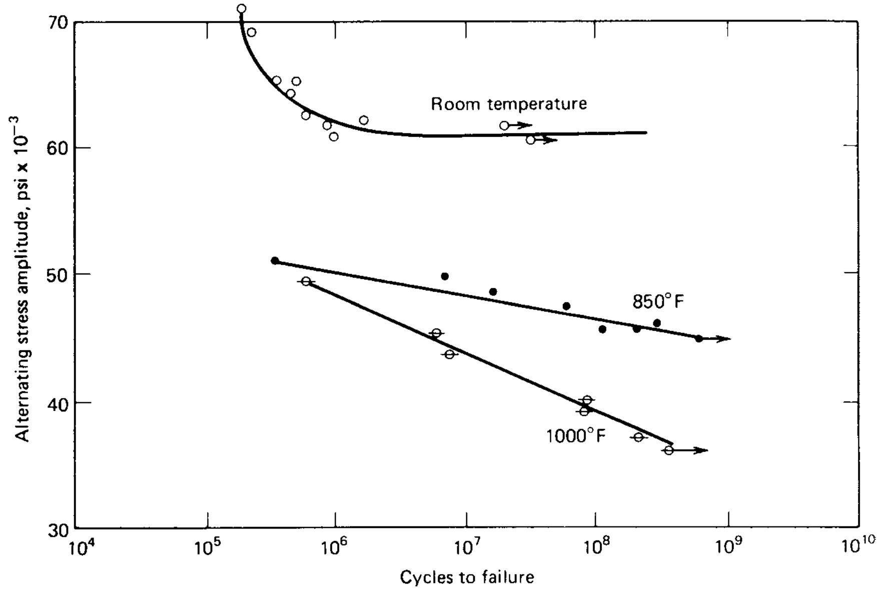


Fig. 18 Effect of operating temperature on the S–N curve of a 12% chromium steel alloy. (Data from Ref. 22)

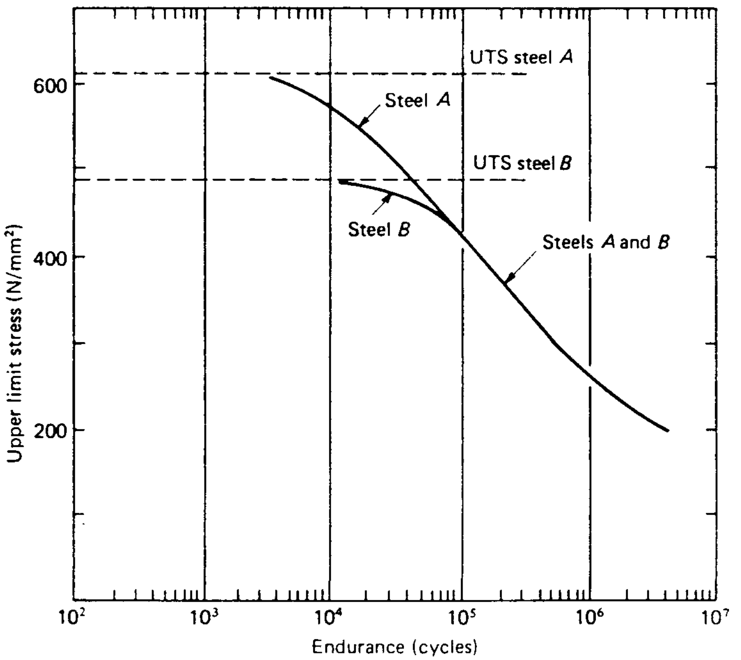


Fig. 19 Effect of ultimate strength on the *S–N* curve for transverse butt welds in two steels. (Data from Ref. 23.)

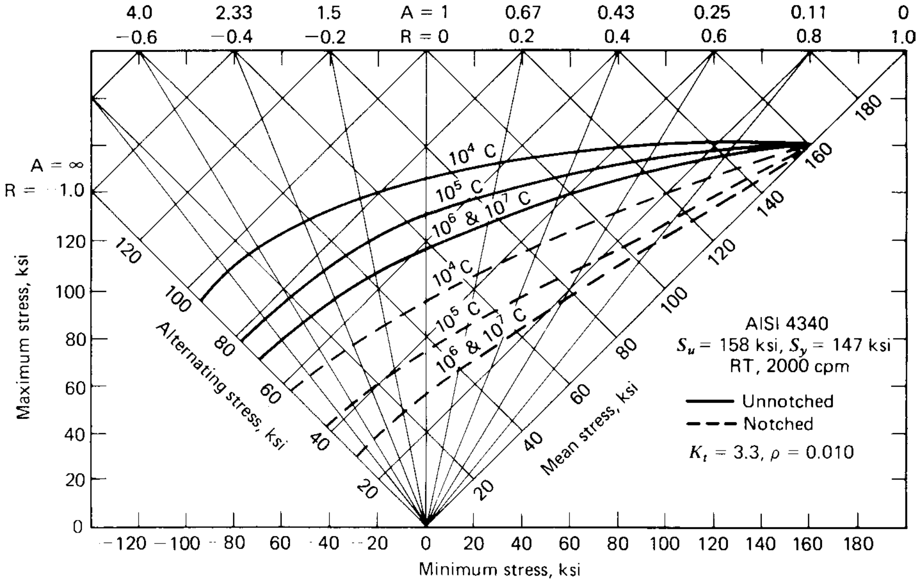


Fig. 20 Master diagram for 4340 steel. (From Ref. 24, p. 37.)

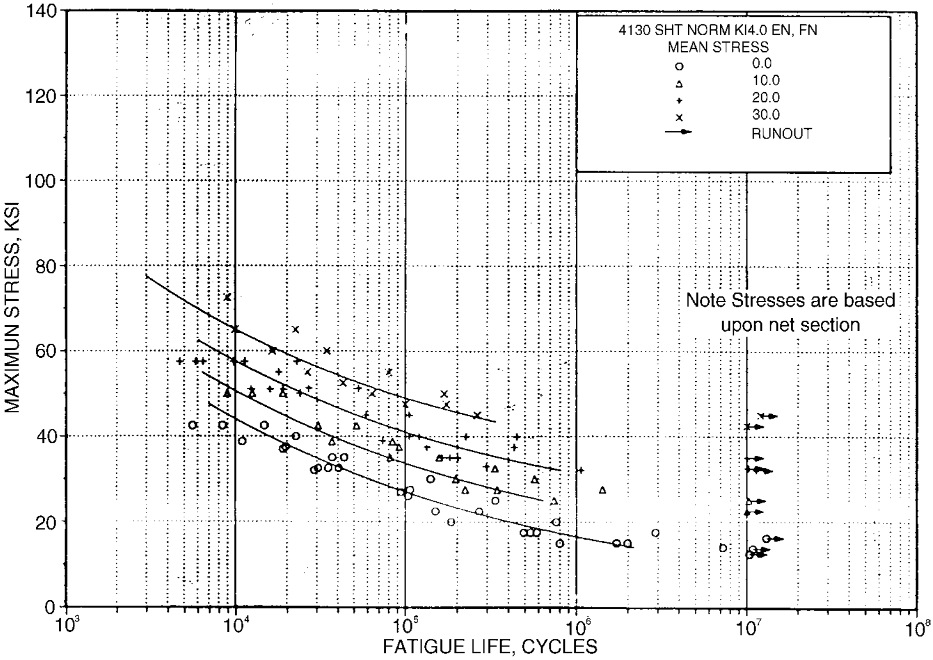


Fig. 21 Best-fit S–N curves for notched 4130 alloy steel sheet, $K_t = 4.0$. (From Ref. 10.)

failure at a given life under nonzero mean conditions to failure at the same life under zero mean cyclic stresses. Historically, the plot of alternating stress amplitude σ_a versus mean stress σ_m has been the object of numerous empirical curve-fitting attempts. The more successful attempts have resulted in four different relationships:

1. Goodman's linear relationship
2. Gerber's parabolic relationship
3. Soderberg's linear relationship
4. The elliptic relationship

A modified form of the Goodman relationship is recommended for general use under conditions of high-cycle fatigue. For tensile mean stress ($\sigma_m > 0$), this relationship may be written

$$\frac{\sigma_a}{\sigma_N} + \frac{\sigma_m}{\sigma_u} = 1 \qquad (13)$$

where σ_u is the material ultimate strength and σ_N is the zero mean stress fatigue strength for a given number of cycles N. For a given alternating stress, compressive mean stresses ($\sigma_m < 0$) have been empirically observed to increase fatigue resistance. However, for conservatism, it is typically assumed that compressive mean stress exerts no influence on fatigue life. Thus, for $\sigma_m < 0$, the fatigue response is identical to that for $\sigma_m = 0$ with $\sigma_a = \sigma_N$.

The modified Goodman relationship is illustrated in Fig. 22. This curve is a failure locus for the case of *uniaxial* fatigue stressing. Any cyclic loading that produces an alternating stress and mean stress that exceeds the bounds of the locus will cause failure in fewer than N cycles. Any alternating stress–mean stress combination that lies within the locus will result in more than N cycles without failure. Combinations on the locus produce failure in N cycles. The modified Goodman relationship shown in Fig. 22 considers fatigue failure exclusively. The reader is cautioned to ensure that the maximum and minimum stresses produced by the cyclic loading do not exceed the material yield strength σ_{yp} such that failure by yielding would be predicted to occur.

5.5 Fatigue Crack Propagation

A fatigue crack that has been initiated by cyclic loading, or any other preexisting flaw in the structure or material, may be expected to grow under sustained cyclic loading until it reaches the critical size from which it will propagate rapidly to catastrophic failure in accordance with the principles of fracture mechanics. For many structures or machine elements, the time required for a fatigue-initiated crack or a preexisting flaw to grow to critical size is a significant portion of the total life.

The fatigue crack growth rate da/dN has been found to often correlate with the crack-tip stress intensity factor range such that

$$\frac{da}{dN} = g(\Delta K) \tag{14}$$

where ΔK is the mode I stress intensity factor range, computed using the maximum and minimum applied stresses with $\Delta K = K_{\max} - K_{\min}$. Most crack growth rate data produced have been characterized in terms of ΔK. For example, Fig. 23 illustrates indirectly the dependence of fatigue crack growth on stress intensity factor. The crack growth rate, indicated by the slope of the a versus N curves, increases with both the applied load and crack length. Since the crack-tip stress intensity factor range also increases with applied load and crack length, it is clear that the crack growth rate is related to the applied stress intensity factor range.

To plot the data of Fig. 23 in terms of the stress intensity factor range and crack growth rate, the crack growth rate is estimated from a numerically deter-

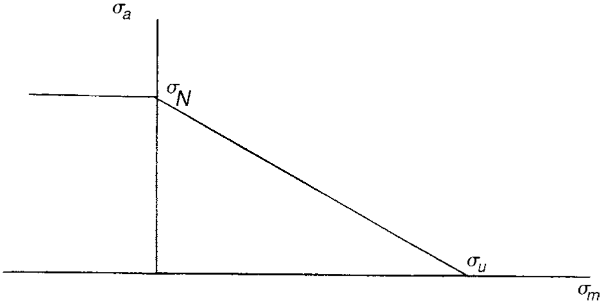


Fig. 22 Modified Goodman relationship.

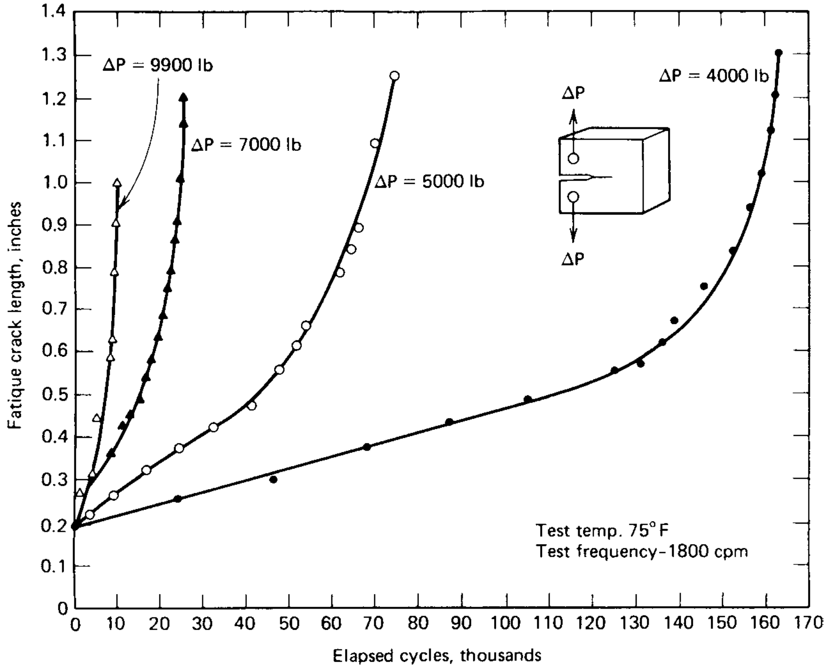


Fig. 23 Effect of cyclic-load range on crack growth in Ni–Mo–V alloy steel for released tension loading. (From Ref. 37. Reprinted with permission of the Society for Experimental Mechanics, www.sem.org.)

mined slope of the a versus N curves between successive data points. Corresponding values of ΔK are then computed from the applied load range and mean crack length for each interval. The results of this procedure are shown in Fig. 24 for the data presented in Fig. 23. It should be noted that all the curves of Fig. 23 are incorporated into the single curve shown in Fig. 24 through use of the stress intensity factor, and the curve of Fig. 24 is therefore applicable to any combination of cyclic stress range and crack length for released loading ($R = 0$) on specimens of this geometry. Different geometries under different applied stresses will exhibit identical crack-tip stress fields if the stress intensity factors are equal. Thus, because the stress intensity factor characterizes the state of stress near the crack tip, the fatigue crack growth rate correlation shown in Fig. 24 is applicable to any cyclically loaded component with $R = 0$ manufactured using the same material. This allows crack growth data generated from simple laboratory specimens to be utilized for approximate crack growth predictions in more complex geometries.

Fatigue crack growth rate data similar to that shown in Fig. 24 have been reported for a wide variety of engineering metals. The linear behavior observed using log-log coordinates suggests that Eq. (14) may be generalized as follows:

$$\frac{da}{dN} = C(\Delta K)^n \tag{15}$$

where n is the slope of the log da/dN versus log ΔK plot and C is the da/dN

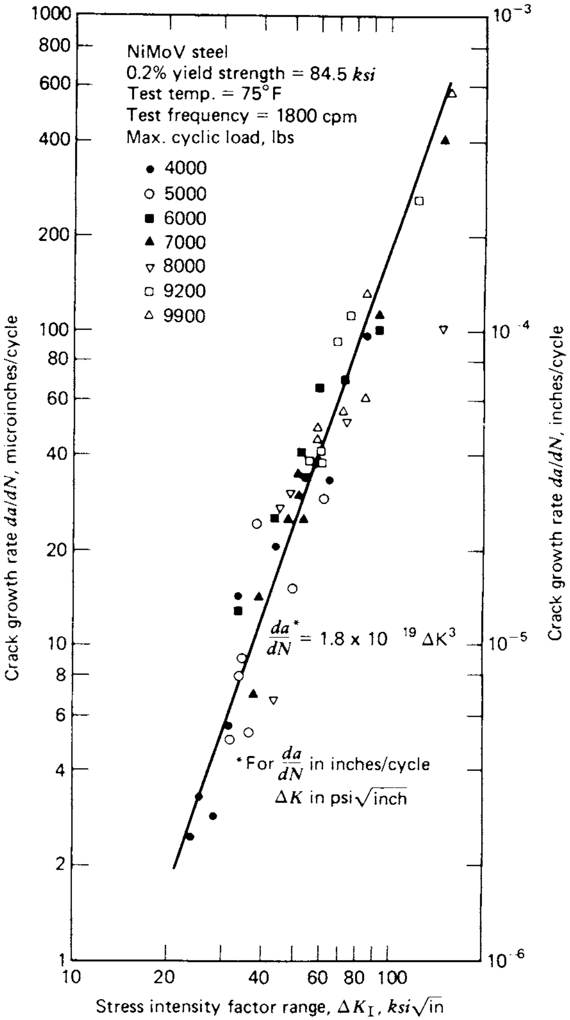


Fig. 24 Crack growth rate as a function of stress–intensity range for Ni–Mo–V steel. (From Ref. 37. Reprinted with permission of the Society for Experimental Mechanics, www.sem.org.)

value found by extending the straight line to a ΔK value of unity. This relationship was first proposed by Paris.[27] The empirical parameters C and n are a function of material, R ratio, thickness, temperature, environment, and loading frequency. Standard methods have been established for conducting fatigue crack growth tests,[25] and fatigue crack growth rate data may be found in Ref. 9, 10, and 12–15. Many other fracture mechanics-based empirical correlations other than Eq. (15) have been proposed, some of which are discussed by Schijve.[26] An extensive overview of the fatigue crack propagation problem is provided by Pook.[32]

Given an initial crack of length a_i, Eq. (15) may be integrated to give the number of cycles N required to propagate a crack to a size a_N such that

$$N = \int_{a_i}^{a_N} \frac{da}{C(\Delta K)^n} \tag{16}$$

Given that ΔK is a function of crack length a, numerical integration techniques will in general be required to compute N. An approximate procedure and several idealized examples are presented by Parker.[35]

It must be emphasized that Eqs. (15) and (16) are applicable only to region II crack growth, as illustrated in Fig. 25. Region I of Fig. 25 exhibits a threshold ΔK_{th} below which the crack will not propagate. Region III corresponds to the transition into the unstable regime of rapid crack extension. In this region, crack growth rates are large and the number of cycles associated with growth in this region small.

With an initial crack of length a_i, from Eq. (16), the number of cycles required to grow a crack to a critical length a_c such that rapid crack extension would be predicted may be approximated as

$$N_p = \int_{a_i}^{a_c} \frac{da}{C(\Delta K)^n} \tag{17}$$

Assuming an initial crack that has been initiated by cyclic loading, the crack propagation life N_p given by Eq. (17) may then be added to the crack initiation life N_i to obtain an estimate of the total fatigue life N with

$$N = N_i + N_p \tag{18}$$

Such estimates are highly sensitive to the length of the initial crack a_i. While

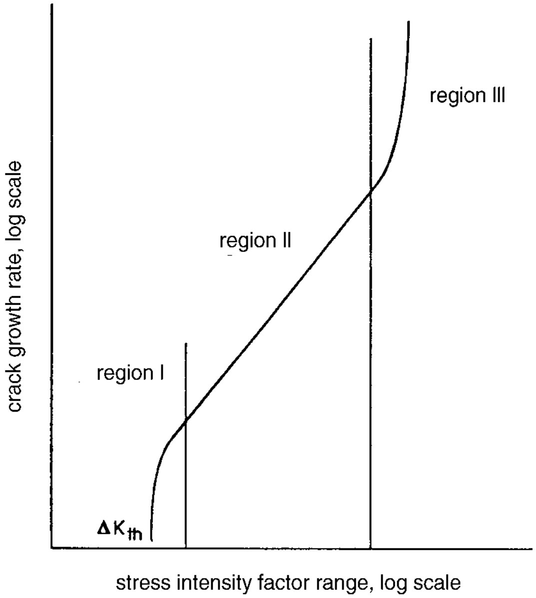


Fig. 25 Schematic representation of fatigue crack growth rate data.

the local stress–strain approach[1,33] may be used to compute the number of cycles N_i required to initiate a crack, the corresponding length of this initiated crack is not defined specifically. No consensus has yet been reached regarding the length of this initiated crack. A size of between 0.25 and 5.0 mm has been suggested,[33] as cracks of this size normally exist at fracture in the small laboratory specimens used to generate the strain versus cycles to failure data required for the local stress–strain approach. An alternative approach would involve the assumption of a preexisting material or manufacturing defect such that $N_i \approx 0$. For example, such an assumption is often made during the analysis of welded joints.[28] If nondestructive techniques are employed, a reasonable assumption for the size of this initial defect would be the largest flaw that could avoid detection.

Crack growth rates determined from constant-amplitude cyclic loading tests are approximately the same as for random loading tests in which the maximum stress is held constant but mean and range of stress vary randomly. However, in random loading tests where the maximum stress is also allowed to vary, the sequence of loading cycles may have a marked effect on crack growth rate, with the overall crack growth being significantly higher for random loading spectra.

Many investigations have shown a significant delay in crack propagation following intermittent application of high stresses. That is, fatigue damage and crack extension are dependent on preceding cyclic load history. This dependence of crack extension on preceding history and the effects upon future damage increments are referred to as *interaction* effects. Most of the interaction studies conducted have dealt with *retardation* of crack growth as a result of the application of occasional tensile overload cycles. Retardation may be characterized as a period of reduced crack growth rate following the application of a peak load or loads higher and in the same direction as those peaks that follow.

The modeling of interaction effects requires consideration of crack-tip plasticity and its subsequent influence. In metals of all types, cracks will remain closed or partially closed for a portion of the applied cyclic load as a consequence of plastically deformed material left in the wake of the growing crack. Under cyclic loading, crack growth will occur during the loading portion of the cycle. Given that a plastic zone exists at the crack tip prior to crack extension, as the material at the crack tip separates, the newly formed crack surfaces will exhibit a layer of plastically deformed material along the newly formed crack faces. Subsequent unloading will compress this plastically deformed material, closing the crack while the applied stress remains tensile. This phenomenon is known as plasticity-induced fatigue crack closure and was first discussed by Elber.[30] Upon reloading during the following cycle, crack growth will not continue unless the applied load is sufficiently large such that the compressive stresses acting along the crack surfaces are overcome and the crack is fully opened. This load is known as the crack opening load and has been demonstrated to be a key parameter in determining fatigue crack growth rates under both constant amplitude and spectrum loading. Further information regarding crack closure may be found in Refs. 1, 4, 16, 26, and 31.

Discussion to this point has been limited to the growth of through-thickness cracks under mode I loading. While mode I loading is often dominant, under the most general circumstances the applied cyclic loads will generate stress intensity factor ranges ΔK_{I}, ΔK_{II}, and ΔK_{III} at the crack tip, and *mixed-mode* fatigue crack growth must be considered. Modeling methodologies for mixed-mode fatigue crack growth are discussed in Refs. 34 and 35. In addition, fatigue cracks in machine elements and structures are often not through-thickness cracks but rather surface cracks that extend partially through the thickness. Such surface cracks are often semielliptical in shape and the analysis of these cracks is considerably more complicated. Information regarding surface cracks may be found in Refs. 4 and 29.

Research has suggested that when fatigue cracks are small, crack growth rates are larger than would be predicted using Eq. (15) for a given ΔK.[36] Small-crack behavior is often important, as a significant portion of the fatigue life may be spent in the small-crack regime. The fatigue crack propagation life N_p will also be influenced by the presence of residual stresses such as might exist as a consequence of welding, heat treatment, carburizing, grinding, or shot-peening. Compressive residual stresses are beneficial, decreasing the rate of fatigue crack growth and increasing propagation life. While approximate methodologies exist for incorporating the effects of residual stress within fatigue crack growth predictions,[35] residual stress distributions are often difficult to characterize.

Reasonable design estimates for the fatigue crack propagation life may be obtained using Eq. (17). However, the many uncertainties typically associated with fatigue life predictions emphasize the essential requirement to conduct full-scale fatigue tests to provide acceptable reliability.

6 CREEP AND STRESS RUPTURE

Creep in its simplest form is the progressive accumulation of plastic strain in a specimen or machine part under stress at elevated temperature over a period of time. Creep failure occurs when the accumulated creep strain results in a deformation of the machine part that exceeds the design limits. *Creep rupture* is an extension of the creep process to the limiting condition where the stressed member actually separates into two parts. *Stress rupture* is a term used interchangeably by many with creep rupture; however, others reserve the term stress rupture for the rupture termination of a creep process in which steady-state creep is never reached, and use the term creep rupture for the rupture termination of a creep process in which a period of steady-state creep has persisted. Figure 26 illustrates these differences. The interaction of creep and stress rupture with cyclic stressing and the fatigue process has not yet been clearly understood but is of great importance in many modern high-performance engineering systems.

Creep strains of engineering significance are not usually encountered until the operating temperatures reach a range of approximately 35–70% of the melting point on a scale of absolute temperature. The approximate melting temperature for several substances is shown in Table 2.

Not only is excessive deformation due to creep an important consideration, but other consequences of the creep process may also be important. These might include creep rupture, thermal relaxation, dynamic creep under cyclic loads or cyclic temperatures, creep and rupture under multiaxial states of stress, cumulative creep effects, and effects of combined creep and fatigue.

Creep deformation and rupture are initiated in the grain boundaries and proceed by sliding and separation. Thus, creep rupture failures are intercrystalline, in contrast, for example, to the transcrystalline failure surface exhibited by room temperature fatigue failures. Although creep is a plastic flow phenomenon, the

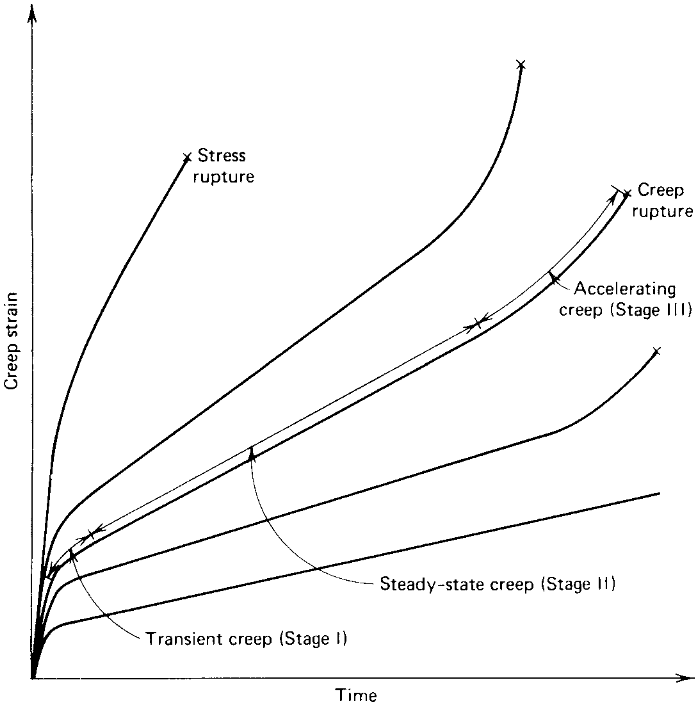


Fig. 26 Illustration of creep and stress rupture.

Table 2 Melting Temperatures[38]

Material	°F	°C
Hafnium carbide	7030	3887
Graphite (sublimes)	6330	3500
Tungsten	6100	3370
Tungsten carbide	5190	2867
Magnesia	5070	2800
Molybdenum	4740	2620
Boron	4170	2300
Titanium	3260	1795
Platinum	3180	1750
Silica	3140	1728
Chromium	3000	1650
Iron	2800	1540
Stainless steels	2640	1450
Steel	2550	1400
Aluminum alloys	1220	660
Magnesium alloys	1200	650
Lead alloys	605	320

intercrystalline failure path gives a rupture surface that has the appearance of brittle fracture. Creep rupture typically occurs without necking and without warning. Current state-of-the-art knowledge does not permit a reliable prediction of creep or stress rupture properties on a theoretical basis. Furthermore, there seems to be little or no correlation between the creep properties of a material and its room temperature mechanical properties. Therefore, test data and empirical methods of extending these data are relied on heavily for prediction of creep behavior under anticipated service conditions.

Metallurgical stability under long-time exposure to elevated temperatures is mandatory for good creep-resistant alloys. Prolonged time at elevated temperatures acts as a tempering process, and any improvement in properties originally gained by quenching may be lost. Resistance to oxidation and other corrosive media are also usually important attributes for a good creep-resistant alloy. Larger grain size may also be advantageous since this reduces the length of grain boundary, where much of the creep process resides.

6.1 Prediction of Long-Term Creep Behavior

Much time and effort has been expended in attempting to develop good short-time creep tests for accurate and reliable prediction of long-term creep and stress rupture behavior. It appears, however, that really reliable creep data can be obtained only by conducting long-term creep tests that duplicate actual service loading and temperature conditions as nearly as possible. Unfortunately, designers are unable to wait for years to obtain design data needed in creep failure analysis. Therefore, certain useful techniques have been developed for approximating long-term creep behavior based on a series of short-term tests. Data from creep testing may be plotted in a variety of different ways. The basic variables involved are stress, strain, time, temperature, and, perhaps, strain rate. Any two of these basic variables may be selected as plotting coordinates, with the remaining variables treated as parametric constants for a given curve. Three commonly used methods for extrapolating short-time creep data to long-term applications are the abridged method, the mechanical acceleration method, and the thermal acceleration method. In the abridged method of creep testing the tests are conducted at several different stress levels and at the contemplated operating temperature. The data are plotted as creep strain versus time for a family of stress levels, all run at constant temperature. The curves are plotted out to the laboratory test duration and then extrapolated to the required design life. In the mechanical acceleration method of creep testing, the stress levels used in the laboratory tests are significantly higher than the contemplated design stress levels, so the limiting design strains are reached in a much shorter time than in actual service. The data taken in the mechanical acceleration method are plotted as stress level versus time for a family of constant strain curves all run at a constant temperature. The thermal acceleration method involves laboratory testing at temperatures much higher than the actual service temperature expected. The data are plotted as stress versus time for a family of constant temperatures where the creep strain produced is constant for the whole plot.

It is important to recognize that such extrapolations are not able to predict the potential of failure by creep rupture prior to reaching the creep design life.

In any testing method it should be noted that creep testing guidelines usually dictate that test periods of less than 1% of the expected life are not deemed to give significant results. Tests extending to at least 10% of the expected life are preferred where feasible.

Several different theories have been proposed to correlate the results of short-time elevated-temperature tests with long-term service performance at more moderate temperatures. One of the more accurate and useful of these proposals is the Larson–Miller theory.

The Larson–Miller theory[39] postulates that for each combination of material and stress level there exists a unique value of a parameter P that is related to temperature and time by the equation

$$P = (\theta + 460)(C + \log_{10} t) \tag{19}$$

where P = Larson–Miller parameter, constant for a given material and stress level
θ = temperature, °F
C = constant, usually assumed to be 20
t = time in hours to rupture or to reach a specified value of creep strain

This equation was investigated for both creep and rupture for some 28 different materials by Larson and Miller with good success. By using (19) it is a simple matter to find a short-term combination of temperature and time that is equivalent to any desired long-term service requirement. For example, for any given material at a specified stress level the test conditions listed in Table 3 should be equivalent to the operating conditions.

6.2 Creep under a Uniaxial State of Stress

Many relationships have been proposed to relate stress, strain, time, and temperature in the creep process. If one investigates experimental creep strain versus time data, it will be observed that the data are close to linear for a wide variety of materials when plotted on log strain versus log time coordinates. Such a plot is shown, for example, in Fig. 27 for three different materials. An equation describing this type of behavior is

$$\delta = At^a \tag{20}$$

Table 3 Equivalent Conditions Based on Larson–Miller Parameter

Operating Condition	Equivalent Test Condition
10,000 h at 1000°F	13 h at 1200°F
1,000 h at 1200°F	12 h at 1350°F
1,000 h at 1350°F	12 h at 1500°F
1,000 h at 300°F	2.2 h at 400°F

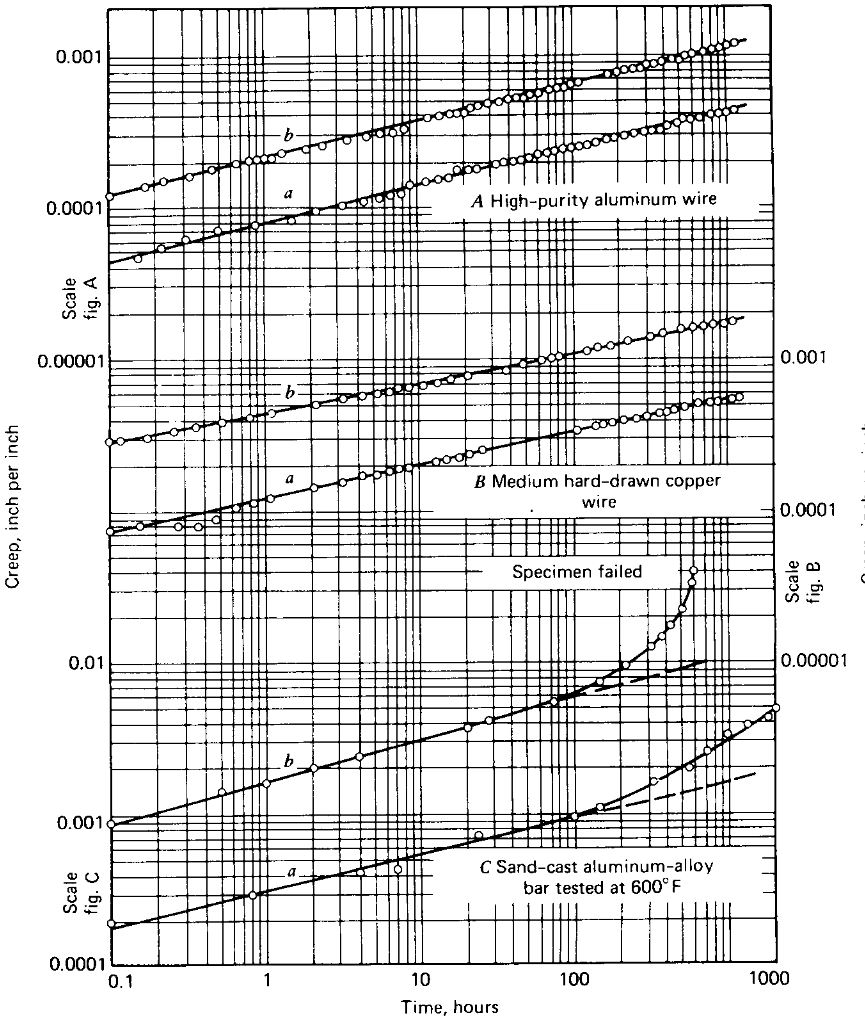


Fig. 27 Creep curves for three materials. (From Ref. 40.)

where δ = true creep strain
t = time
A, a = empirical constants

Differentiating (20) with respect to time gives

$$\dot{\delta} = bt^{-n} \tag{21}$$

or, setting $aA = b$ and $1 - a = n$,

$$\dot{\delta} = bt^{-n} \tag{22}$$

This equation represents a variety of different types of creep strain versus time curves, depending on the magnitude of the exponent n. If n is zero, the behavior, characteristic of high temperatures, is termed *constant creep rate*, and the creep strain is given as

$$\delta = b_1 t + C_1 \tag{23}$$

If n lies between 0 and 1, the behavior is termed *parabolic creep,* and the creep strain is given by

$$\delta = b_3 t^m + C_3 \tag{24}$$

This type of creep behavior occurs at intermediate and high temperatures. The coefficient b_3 increases exponentially with stress and temperature, and the exponent m decreases with stress and increases with temperature. The influence of stress level σ on creep rate can often be represented by the empirical expression

$$\dot{\delta} = b\sigma^N \tag{25}$$

Assuming the stress σ to be independent of time, we may integrate (25) to yield the creep strain

$$\delta = Bt\sigma^N + C' \tag{26}$$

If the constant C' is small compared with $Bt\sigma^N$, as it often is, the result is called the *log-log stress–time creep law,* given as

$$\sigma = Bt\sigma^N \tag{27}$$

As long as the instantaneous deformation on load application and the stage I transient creep are small compared to stage II steady-state creep, Eq. (27) is useful as a design tool.

If it is necessary to consider all stages of the creep process, the creep strain expression becomes much more complex. The most general expression for the creep process is[41]

$$\delta = \frac{\sigma}{E} + k_1\sigma^m + k_2(1 - e^{-qt})\sigma^n + k_3 t\sigma^p \tag{28}$$

where δ = total creep strain
σ/E = initial elastic strain
$k_1\sigma^m$ = initial plastic strain
$k_2(1 - e^{-qt})\sigma^n$ = anelastic strain
$k_3 t\sigma^p$ = viscous strain
σ = stress
E = modulus of elasticity
m = reciprocal of strain-hardening exponent
k_1 = reciprocal of strength coefficient

q = reciprocal of Kelvin retardation time
k_2 = anelastic coefficient
n = empirical exponent
k_3 = viscous coefficient
p = empirical exponent
t = time

To utilize this empirical nonlinear expression in a design environment requires specific knowledge of the constants and exponents that characterize the material and temperature of the application. In all cases it must be recognized that stress rupture may intervene to terminate the creep process, and the prediction of this occurrence is difficult.

7 FRETTING AND WEAR

Fretting and wear share many common characteristics but, at the same time, are distinctly different in several ways. Basically, fretting action has, for many years, been defined as a combined mechanical and chemical action in which contacting surfaces of two solid bodies are pressed together by a normal force and are caused to execute oscillatory sliding relative motion, wherein the magnitude of normal force is great enough and the amplitude of the oscillatory sliding motion is small enough to significantly restrict the flow of fretting debris away from the originating site.[42] More recent definitions of fretting action have been broadened to include cases in which contacting surfaces periodically separate and then reengage, as well as cases in which the fluctuating friction-induced surface tractions produce stress fields that may ultimately result in failure. The complexities of fretting action have been discussed by numerous investigators, who have postulated the combination of many mechanical, chemical, thermal, and other phenomena that interact to produce fretting. Among the postulated phenomena are plastic deformation caused by surface asperities plowing through each other, welding and tearing of contacting asperities, shear and rupture of asperities, friction-generated subsurface shearing stresses, dislodging of particles and corrosion products at the surfaces, chemical reactions, debris accumulation and entrapment, abrasive action, microcrack initiation, and surface delamination.[43–58]

Damage to machine parts due to fretting action may be manifested as corrosive surface damage due to fretting corrosion, loss of proper fit or change in dimensions due to fretting wear, or accelerated fatigue failure due to fretting fatigue. Typical sites of fretting damage include interference fits; bolted, keyed, splined, and riveted joints; points of contact between wires in wire ropes and flexible shafts; friction clamps; small-amplitude-of-oscillation bearings of all kinds; contacting surfaces between the leaves of leaf springs; and all other places where the conditions of fretting persist. Thus, the efficiency and reliability of the design and operation of a wide range of mechanical systems are related to the fretting phenomenon.

Wear may be defined as the undesired cumulative change in dimensions caused by the gradual removal of discrete particles from contacting surfaces in motion, due predominantly to mechanical action. It should be further recognized that corrosion often interacts with the wear process to change the character of the surfaces of wear particles through reaction with the environment. Wear is, in fact, not a single process but a number of different processes that may take place by themselves or in combination. It is generally accepted that there are at least five major subcategories of wear (see p. 120 of Ref. 59, see also Ref. 60), including adhesive wear, abrasive wear, corrosive wear, surface fatigue wear, and deformation wear. In addition, the categories of fretting wear and impact wear[55,61,62] have been recognized by wear specialists. Erosion and cavitation are sometimes considered to be categories of wear as well. Each of these types of wear proceeds by a distinctly different physical process and must be separately considered, although the various subcategories may combine their influence either by shifting from one mode to another during different eras in the operational lifetime of a machine or by simultaneous activity of two or more different wear modes.

7.1 Fretting Phenomena

Although fretting fatigue, fretting wear, and fretting corrosion phenomena are potential failure modes in a wide variety of mechanical systems, and much research effort has been devoted to the understanding of the fretting process, there are very few quantitative design data available, and no generally applicable design procedure has been established for predicting failure under fretting conditions. However, even though the fretting phenomenon is not fully understood, and a good general model for prediction of fretting fatigue or fretting wear has not yet been developed, significant progress has been made in establishing an understanding of fretting and the variables of importance in the fretting process. It has been suggested that there may be more than 50 variables that play some role in the fretting process.[63] Of these, however, there are probably only 8 that are of major importance:

1. The magnitude of relative motion between the fretting surfaces
2. The magnitude and distribution of pressure between the surfaces at the fretting interface
3. The state of stress, including magnitude, direction, and variation with respect to time in the region of the fretting surfaces
4. The number of fretting cycles accumulated
5. he material, and surface condition, from which each of the fretting members is fabricated
6. Cyclic frequency of relative motion between the two members being fretted
7. Temperature in the region of the two surfaces being fretted
8. Atmospheric environment surrounding the surfaces being fretted

These variables interact so that a quantitative prediction of the influence of any given variable is very dependent on all the other variables in any specific application or test. Also, the combination of variables that produce a very serious consequence in terms of fretting fatigue damage may be quite different from the combinations of variables that produce serious fretting wear damage. No general techniques yet exist for quantitatively predicting the influence of the important variables of fretting fatigue and fretting wear damage, although many special cases have been investigated. However, it has been observed that certain trends usually exist when the variables just listed are changed. For example, fretting damage tends to increase with increasing contact pressure until a nominal pressure of a few thousand pounds per square inch is reached, and further increases in pressure seem to have relatively little direct effect. The state of stress is important, especially in fretting fatigue. Fretting damage accumulates with increasing numbers of cycles at widely different rates, depending on specific operating conditions. Fretting damage is strongly influenced by the material properties of the fretting pair-surface hardness, roughness, and finish. No clear trends have been established regarding frequency effects on fretting damage, and although both temperature and atmospheric environment are important influencing factors, their influences have not been clearly established. A clear presentation relative to these various parameters is given in Ref. 55.

Fretting fatigue is fatigue damage directly attributable to fretting action. It has been suggested that premature fatigue nuclei may be generated by fretting through either abrasive pit-digging action, asperity-contact microcrack initiation,[64] friction-generated cyclic stresses that lead to the formation of microcracks,[65] or subsurface cyclic shear stresses that lead to surface delamination in the fretting zone.[58] Under the abrasive pit-digging hypothesis, it is conjectured that tiny grooves or elongated pits are produced at the fretting interface by the asperities and abrasive debris particles moving under the influence of oscillatory relative motion. A pattern of tiny grooves would be produced in the fretted region with their longitudinal axes all approximately parallel and in the direction of fretting motion, as shown schematically in Fig. 28.

The asperity-contact microcrack initiation mechanism is postulated to proceed due to the contact force between the tip of an asperity on one surface and another asperity on the mating surface as the surfaces move back and forth. If the initial contact does not shear one or the other asperity from its base, the repeated contacts at the tips of the asperities give rise to cyclic or fatigue stresses in the region at the base of each asperity. It has been estimated[51] that under such conditions the region at the base of each asperity is subjected to large local stresses that probably lead to the nucleation of fatigue microcracks at these sites. As shown schematically in Fig. 29, it would be expected that the asperity-contact mechanism would produce an array of microcracks whose longitudinal axes would be generally perpendicular to the direction of fretting motion.

The friction-generated cyclic stress fretting hypothesis[53] is based on the observation that when one member is pressed against the other and caused to undergo fretting motion, the tractive friction force induces a compressive tangential stress component in a volume of material that lies ahead of the fretting motion, and a tensile tangential stress component in a volume of material that lies behind the fretting motion, as shown in Fig. 30*a*. When the fretting direction is reversed, the tensile and compressive regions change places. Thus, the volume of material adjacent to the contact zone is subjected to a cyclic stress that is postulated to generate a field of microcracks at these sites. Furthermore, the geometrical stress concentration associated with the clamped joint may contribute to microcrack generation at these sites.[54] As shown in Fig. 30*c*, it would be expected that the friction-generated microcrack mechanism would produce an

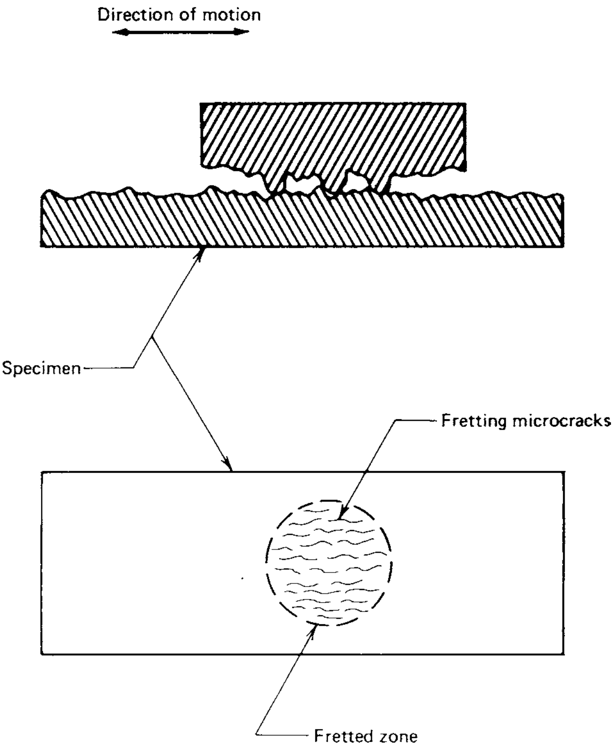


Fig. 28 Idealized schematic illustration of the stress concentrations produced by the abrasive pit-digging mechanism.

array of microcracks whose longitudinal axes would be generally perpendicular to the direction of fretting motion. These cracks would lie in a region adjacent to the fretting contact zone.

In the delamination theory of fretting[58] it is hypothesized that the combination of normal and tangential tractive forces transmitted through the asperity-contact sites at the fretting interface produces a complex multiaxial state of stress, accompanied by a cycling deformation field, which produces subsurface peak shearing stress and subsurface crack nucleation sites. With further cycling, the cracks propagate approximately parallel to the surface, as in the case of the surface fatigue phenomenon, finally propagating to the surface to produce a thin wear sheet, which "delaminates" to become a particle of debris.

Supporting evidence has been generated to indicate that under various circumstances each of the four mechanisms is active and significant in producing fretting damage.

The influence of the state of stress in the member during the fretting is shown for several different cases in Fig. 31, including static tensile and compressive mean stresses during fretting. An interesting observation in Fig. 31 is that fretting under conditions of compressive mean stress, either static or cyclic, produces a drastic reduction in fatigue properties. This, at first, does not seem to be in keeping with the concept that compressive stresses are beneficial in fatigue load-

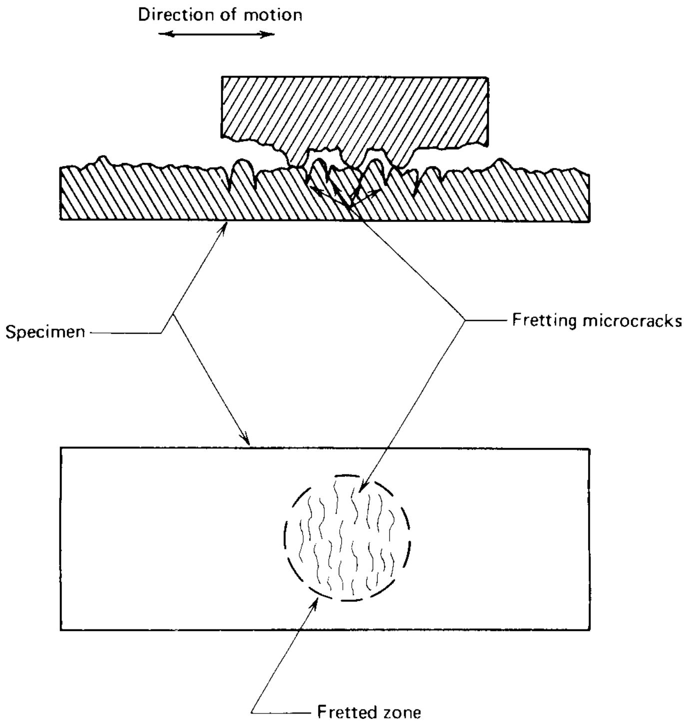


Fig. 29 Idealized schematic illustration of the stress concentrations produced by the asperity-contact microcrack initiation mechanism.

ing. However, it was deduced[66] that the compressive stresses during fretting shown in Fig. 31 actually resulted in local residual tensile stresses in the fretted region. Likewise, the tensile stresses during fretting shown in Fig. 31 actually resulted in local residual compressive stresses in the fretted region. The conclusion, therefore, is that local compressive stresses are beneficial in minimizing fretting fatigue damage.

Further evidence of the beneficial effects of compressive residual stresses in minimizing fretting fatigue damage is illustrated in Fig. 32, where the results of a series of Prot (fatigue limit) tests are reported for steel and titanium specimens subjected to various combinations of shot peening and fretting or cold rolling and fretting. It is clear from these results that the residual compressive stresses produced by shot peening and cold rolling are effective in minimizing the fretting damage. The reduction in scatter of the fretted fatigue properties for titanium is especially important to a designer because design stress is closely related to the lower limit of the scatter band.

In the final analysis, it is necessary to evaluate the seriousness of fretting fatigue damage in any specific design by running simulated service tests on specimens or components. Within the current state-of-the-art knowledge in the area of fretting fatigue, there is no other safe course of action open to the designer.

Fretting wear is a change in dimensions through wear directly attributable to the fretting process between two mating surfaces. It is thought that the abrasive

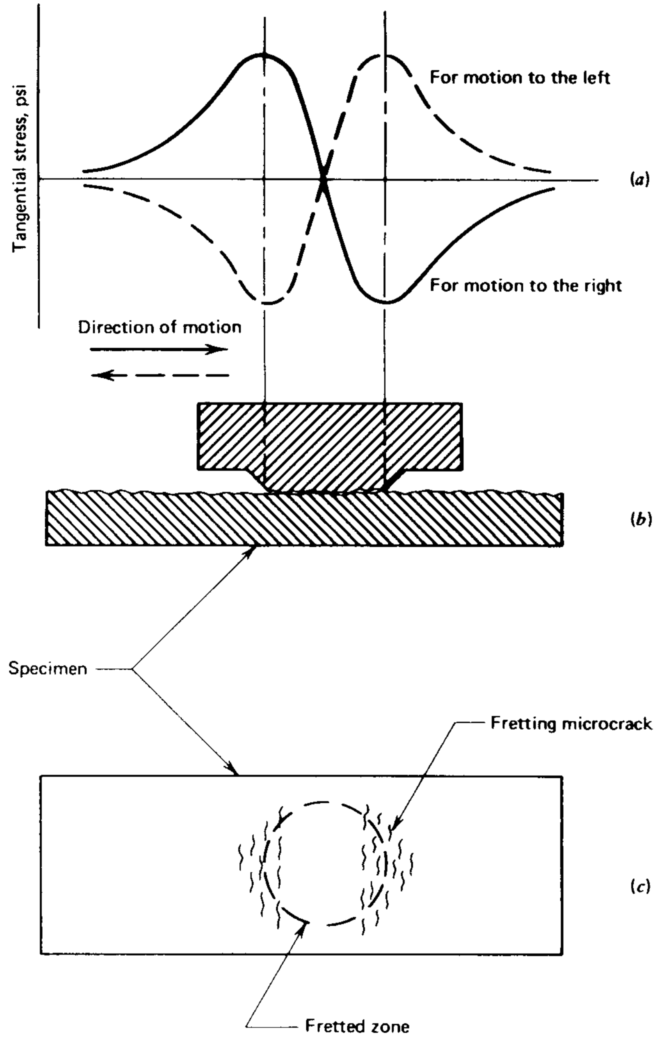


Fig. 30 Idealized schematic illustration of the tangential stress components and microcracks produced by the friction-generated microcrack initiation mechanism.

pit-digging mechanism, the asperity-contact microcrack initiation mechanism, and the wear-sheet delamination mechanism may all be important in most fretting wear failures. As in the case of fretting fatigue, there has been no good model developed to describe the fretting wear phenomenon in a way useful for design. An expression for weight loss due to fretting has been proposed[48] as

$$W_{\text{total}} \; (k_0 L^{1/2} - k_1 L) \frac{C}{F} + k_2 SLC \tag{29}$$

where W_{total} = total specimen weight loss
L = normal contact load
C = number of fretting cycles

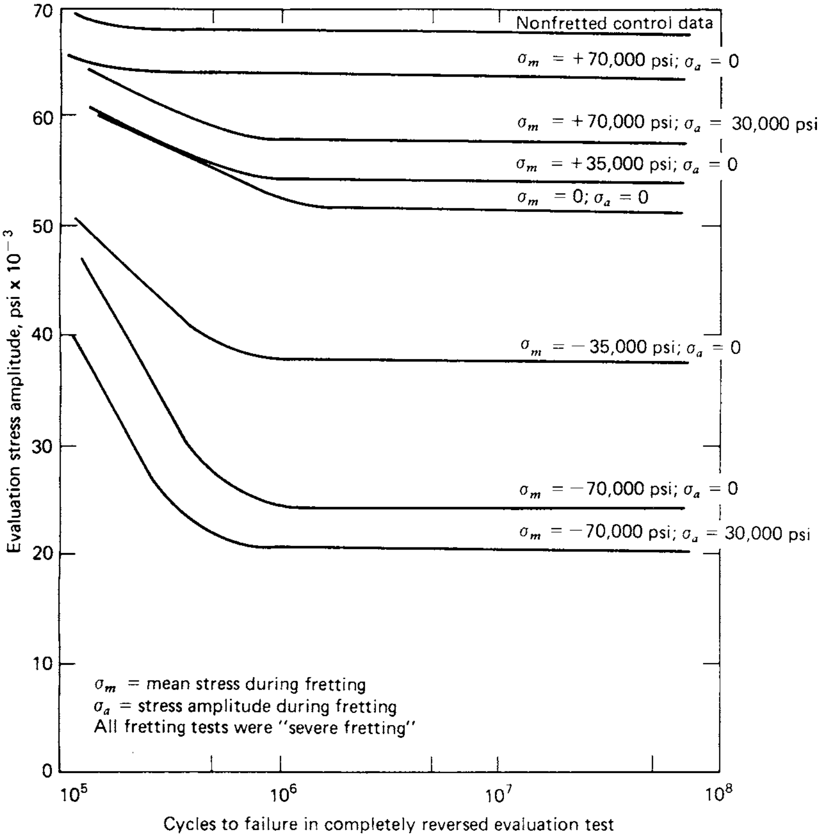


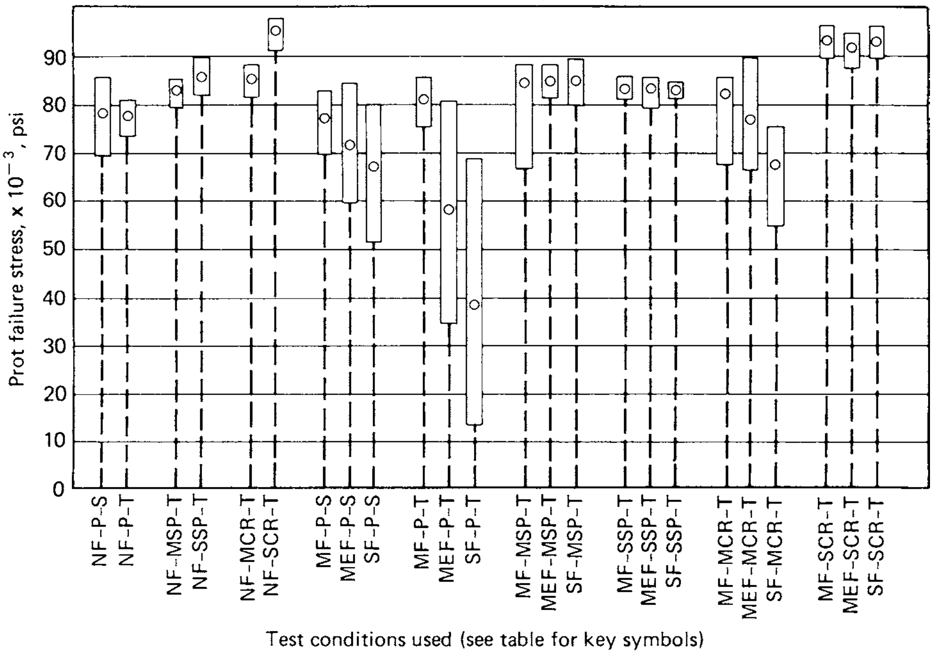
Fig. 31 Residual fatigue properties subsequent to fretting under various states of stress.

F = frequency of fretting
S = peak-to-peak slip between fretting surfaces
k_0, k_1, k_2 = constants to be empirically determined

This equation has been shown to give relatively good agreement with experimental data over a range of fretting conditions using mild steel specimens. However, weight loss is not of direct use to a designer. Wear depth is of more interest. Prediction of wear depth in an actual design application must in general be based on simulated service testing.

Some investigators have suggested that estimates of fretting wear depth may be based on the classical adhesive or abrasive wear equations, in which wear depth is proportional to load and total distance slid, where the total distance slid is calculated by multiplying relative motion per cycle times number of cycles. Although there are some supporting data for such a procedure,[67] more investigation is required before it could be recommended as an acceptable approach for general application.

If fretting wear at a support interface, such as between tubes and support plates of a steam generator or heat exchanger or between fuel pins and support



Test Condition Used	Code Designation	Sample Size	Mean Prot Failure Stress, psi	Unbiased Standard Deviation, psi
Nonfretted, polished, SAE 4340 steel	NF-P-S	15	78,200	5,456
Nonfretted, polished, Ti-140-A titanium	NF-P-T	15	77,800	2,454
Nonfretted, mildly shot-peened, Ti-140-A titanium	NF-MSP-T	15	83,100	1,637
Nonfretted, severely shot-peened, Ti-140-A titanium	NF-SSP-T	15	85,700	2,398
Nonfretted, mildly cold-rolled, Ti-140-A titanium	NF-MCR-T	15	85,430	1,924
Nonfretted, severely cold-rolled, Ti-140-A titanium	NF-SCR-T	15	95,400	2,120
Mildly fretted, polished, SAE 4340 steel	MF-P-S	15	77,280	4,155
Medium fretted, polished, SAE 4340 steel	MeF-P-S	15	71,850	5,492
Severely fretted, polished, SAE 4340 steel	SF-P-S	15	67,700	6,532
Mildly fretted, polished, Ti-140-A titanium	MF-P-T	15	81,050	3,733
Medium fretted, polished, Ti-140-A titanium	MeF-P-T	15	58,140	15,715
Severely fretted, polished, Ti-140-A titanium	SF-P-T	15	38,660	19,342
Mildly fretted, mildly shot-peened, Ti-140-A titanium	MF-MSP-T	15	84,520	5,239
Medium fretted, mildly shot-peened, Ti-140-A titanium	MeF-MSP-T	15	84,930	2,446
Severely fretted, mildly shot-peened, Ti-140-A titanium	SF-MSP-T	15	84,870	2,647
Mildly fretted, severely shot-peened, Ti-140-A titanium	MF-SSP-T	15	83,600	1,474
Medium fretted, severely shot-peened, Ti-140-A titanium	MeF-SSP-T	15	83,240	1,332
Severely fretted, severely shot-peened, Ti-140-A titanium	SF-SSP-T	15	83,110	1,280
Mildly fretted, mildly cold-rolled, Ti-140-A titanium	MF-MCR-T	15	82,050	4,313
Medium fretted, mildly cold-rolled, Ti-140-A titanium	MeF-MCR-T	15	76,930	8,305
Severely fretted, mildly cold-rolled, Ti-140-A titanium	SF-MCR-T	15	67,960	5,682
Mildly fretted, severely cold-rolled, Ti-140-A titanium	MF-SCR-T	15	93,690	1,858
Medium fretted, severely cold-rolled, Ti-140-A titanium	MeF-SCR-T	15	91,950	2,098
Severely fretted, severely cold-rolled, Ti-140-A titanium	SF-SCR-T	15	93,150	1,365

Fig. 32 Fatigue properties of fretted steel and titanium specimens with various degrees of shot peening and cold rolling. (See Ref. 52.)

grids of a reactor core, produces loss of fit at a support site, impact fretting may occur. Impact fretting is fretting action induced by the small lateral relative displacements between two surfaces when they impact together, where the small displacements are caused by Poisson strains or small tangential "glancing" velocity components. Impact fretting has only recently been addressed in the literature,[68] but it should be noted that under certain circumstances impact fretting may be a potential failure mode of great importance.

Fretting corrosion may be defined as any corrosive surface involvement resulting as a direct result of fretting action. The consequences of fretting corrosion are generally much less severe than for either fretting wear or fretting fatigue. Note that the term *fretting corrosion* is not being used here as a synonym for fretting, as in much of the early literature on this topic. Perhaps the most important single parameter in minimizing fretting corrosion is proper selection of the material pair for the application. Table 4 lists a variety of material pairs grouped according to their resistance to fretting corrosion.[69] Cross comparisons from one investigator's results to another's must be made with care because testing conditions varied widely. The minimization or prevention of fretting damage must be carefully considered as a separate problem in each individual design application because a palliative in one application may significantly accelerate fretting damage in a different application. For example, in a joint that is designed to have no relative motion, it is sometimes possible to reduce or prevent fretting by increasing the normal pressure until all relative motion is arrested. However, if the increase in normal pressure does not completely arrest the relative motion, the result may be significantly increasing fretting damage instead of preventing it.

Nevertheless, there are several basic principles that are generally effective in minimizing or preventing fretting. These include:

1. Complete separation of the contacting surfaces.
2. Elimination of all relative motion between the contacting surfaces.
3. If relative motion cannot be eliminated, it is sometimes effective to superpose a large unidirectional relative motion that allows effective lubrication. For example, the practice of driving the inner or outer race of an oscillatory pivot bearing may be effective in eliminating fretting.
4. Providing compressive residual stresses at the fretting surface; this may be accomplished by shot peening, cold rolling, or interference fit techniques.
5. Judicious selection of material pairs.
6. Use of interposed low-shear-modulus shim material or plating, such as lead, rubber, or silver.
7. Use of surface treatments or coatings as solid lubricants.
8. Use of surface grooving or roughening to provide debris escape routes and differential strain matching through elastic action.

Of all these techniques, only the first two are completely effective in preventing fretting. The remaining concepts, however, may often be used to minimize fretting damage and yield an acceptable design.

Table 4 Fretting Corrosion Resistance of Various Material Pairs[69]

	Material Pairs Having Good Fretting Corrosion Resistance		
Sakmann and Rightmire	Lead	on	Steel
	Silver plate	on	Steel
	Silver plate	on	Silver plate
	"Parco-lubrized" steel	on	Steel
Gray and Jenny	Grit blasted steel plus lead plate	on	Steel (very good)
	$\frac{1}{16}$ in. nylon insert	on	Steel (very good)
	Zinc and iron phosphated (Bonderizing) steel	on	Steel (good with thick coat)
McDowell	Laminated plastic	on	Gold plate
	Hardtool steel	on	Tool steel
	Cold-rolled steel	on	Cold-rolled steel
	Cast iron	on	Cast iron with phosphate coating
	Cast iron	on	Cast iron with rubber cement
	Cast iron	on	Cast iron with tungsten sulfide coating
	Cast iron	on	Cast iron with rubber insert
	Cast iron	on	Cast iron with Molykote lubricant
	Cast iron	on	Stainless steel with Molykote lubricant
	Material Pairs Having Intermediate Fretting Corrosion Resistance		
Sakmann and Rightmire	Cadmium	on	Steel
	Zinc	on	Steel
	Copper alloy	on	Steel
	Zinc	on	Aluminum
	Copper plate	on	Aluminum
	Nickel plate	on	Aluminum
	Silver plate	on	Aluminum
	Iron plate	on	Aluminum
Gray and Jenny	Sulfide-coated bronze	on	Steel
	Cast bronze	on	"Parco-lubrized" steel
	Magnesium	on	"Parco-lubrized" steel
	Grit-blasted steel	on	Steel
McDowell	Cast iron	on	Cast iron (rough or smooth surface)
	Copper	on	Cast iron
	Brass	on	Cast iron
	Zinc	on	Cast iron
	Cast iron	on	Silver plate
	Cast iron	on	Copper plate
	Magnesium	on	Copper plate
	Zirconium	on	Zirconium
Sakmann and Rightmire	Steel	on	Steel
	Nickel	on	Steel
	Aluminum	no	Steel
	Al–Si alloy	on	Steel
	Antimony plate	on	Steel
	Tin	on	Steel
	Aluminum	on	Aluminum
	Zinc plate	on	Aluminum
Gray and Jenny	Grit blast plus silver plate	on	Steel[a]
	Steel	on	Steel
	Grit blast plus copper plate	on	Steel
	Grit blast plus in plate	on	Steel
	Grit blast and aluminum foil	on	Steel
	Be–Cu insert	on	Steel
	Magnesium	on	Steel
	Nitrided steel	on	Chromium-plated steel[b]

Table 4 (*Continued*)

	Material Pairs Having Poor Fretting Corrosion Resistance		
McDowell	Aluminum	on	Cast iron
	Aluminum	on	Stainless steel
	Magnesium	on	Cast iron
	Cast iron	on	Chromium plate
	Laminated plastic	on	Cast iron
	Bakelite	on	Cast iron
	Hard tool steel	on	Stainless steel
	Chromium plate	on	Chromium plate
	Cast iron	on	Tin plate
	Gold plate	on	Gold plate

[a] Possibly effective with light loads and thick (0.005 in.) silver plate.
[b] Some improvement by heating chromium plated steel to 538°C for 1 hr.

7.2 Wear Phenomena

The complexity of the wear process may be better appreciated by recognizing that many variables are involved, including the hardness, toughness, ductility, modulus of elasticity, yield strength, fatigue properties, and structure and composition of the mating surfaces, as well as geometry, contact pressure, temperature, state of stress, stress distribution, coefficient of friction, sliding distance, relative velocity, surface finish, lubricants, contaminants, and ambient atmosphere at the wearing interface. Clearance versus contact-time history of the wearing surfaces may also be an important factor in some cases. Although the wear processes are complex, progress has been made toward development of quantitative empirical relationships for the various subcategories of wear under specified operating conditions.

Adhesive wear is often characterized as the most basic or fundamental subcategory of wear since it occurs to some degree whenever two solid surfaces are in rubbing contact and remains active even when all other modes of wear have been eliminated. The phenomenon of adhesive wear may be best understood by recalling that all real surfaces, no matter how carefully prepared and polished, exhibit a general waviness upon which is superposed a distribution of local protuberances or asperities. As two surfaces are brought into contact, therefore, only a relatively few asperities actually touch, and the *real* area of contact is only a small fraction of the *apparent* contact area. (See Chap. 1 of Ref. 44 and Chap. 2 of Ref. 70.) Thus, even under very small applied loads, the local pressures at the contact sites become high enough to exceed the yield strength of one or both surfaces, and local plastic flow ensues. If the contacting surfaces are clean and uncorroded, the very intimate contact generated by this local plastic flow brings the atoms of the two contacting surfaces close enough together to call into play strong adhesive forces. This process is sometimes called *cold welding*. Then if the surfaces are subjected to relative sliding motion, the cold-welded junctions must be broken. Whether they break at the original interface or elsewhere within the asperity depends on surface conditions, temperature distribution, strain-hardening characteristics, local geometry, and stress distribution. If the junction is broken away from the original interface, a particle of one surface is transferred to the other surface, marking one event in the adhesive wear process. Later sliding interactions may dislodge the transferred particles as loose wear particles, or they may remain attached. If this adhesive wear process

Table 5 Archard Adhesive Wear Constant *k* for Various Unlubricated Material Pairs in Sliding Contact

Material	Wear Constant *k*
Zinc on zinc	160×10^{-3}
Low-carbon steel on low-carbon steel	45×10^{-3}
Copper on copper	32×10^{-3}
Stainless steel on stainless steel	21×10^{-3}
Copper (on low-carbon steel)	1.5×10^{-3}
Low-carbon steel (on copper)	0.5×10^{-3}
Bakelite on bakelite	0.02×10^{-3}

Source: From Ref. 71, with permission of John Wiley & Sons.

becomes severe and large-scale metal transfer takes place, the phenomenon is called *galling*. If the galling becomes so severe that two surfaces adhere over a large region so that the actuating forces can no longer produce relative motion between them, the phenomenon is called *seizure*. If properly controlled, however, the adhesive wear rate may be low and self-limiting, often being exploited in the "wearing-in" process to improve mating surfaces such as bearings or cylinders so that full film lubrication may be effectively used.

One quantitative estimate of the amount of adhesive wear is given as follows (see Ref. 59 and Chaps. 2 and 6 of Ref. 71):

$$d_{\text{adh}} = \frac{V_{\text{adh}}}{A_a} = \left(\frac{k}{9\sigma_{\text{yp}}}\right)\left(\frac{W}{A_a}\right) L_s \tag{30}$$

or

$$d_{\text{adh}} = k_{\text{adh}} p_m L_s \tag{31}$$

where d_{adh} is the average wear depth, A_a is the apparent contact area, L_s is the total sliding distance, V_{adh} is the wear volume, W is the applied load, $p_m = W/A_a$ is the mean nominal contact pressure between bearing surfaces, and $k_{\text{adh}} = k/9\sigma_{\text{yp}}$ is a wear coefficient that depends on the probability of formation of a transferred fragment and the yield strength (or hardness) of the softer material. Typical values of the wear constant k for several material pairs are shown in Table 5, and the influence of lubrication on the wear constant k is indicated in Table 6. Noting from (31) that

Table 6 Order of Magnitude Values for Adhesive Wear Constant *k* under Various Conditions of Lubrication

	Metal (on Metal)		Nonmetal
Lubrication Condition	Like	Unlike	(on Metal)
Unlubricated	5×10^{-3}	2×10^{-4}	5×10^{-6}
Poorly lubricated	2×10^{-4}	2×10^{-2}	5×10^{-6}
Average lubrication	2×10^{-5}	2×10^{-5}	5×10^{-6}
Excellent lubrication	2×10^{-6} to 10^{-7}	2×10^{-6} to 10^{-7}	2×10^{-6}

Source: From Ref. 71, with permission of John Wiley & Sons.

$$k_{\text{adh}} = \frac{d_{\text{adh}}}{p_m L_s} \tag{32}$$

it may be observed that if the ratio $d_{\text{adh}}/p_m L_s$ is experimentally found to be constant, Eq. (31) should be valid. Experimental evidence has been accumulated (see pp. 124–125 of Ref. 59) to confirm that for a given material pair this ratio is constant up to mean nominal contact pressures approximately equal to the uniaxial yield strength. Above this level the adhesive wear coefficient increases rapidly, with attendant severe galling and seizure.

In the selection of metal combinations to provide resistance to adhesive wear, it has been found that the sliding pair should be composed of mutually insoluble metals and that at least one of the metals should be from the B subgroup of the periodic table. (See p. 31 of Ref. 72.) The reasons for these observations are that the number of cold-weld junctions formed is a function of the mutual solubility, and the strength of the junction bonds is a function of the bonding characteristics of the metals involved. The metals in the B subgroup of the periodic table are characterized by weak, brittle covalent bonds. These criteria have been verified experimentally, as shown in Table 7, where 114 of 123 pairs tested substantiated the criteria.

In the case of abrasive wear, the wear particles are removed from the surface by the plowing and gouging action of the asperities of a harder mating surface or by hard particles trapped between the rubbing surfaces. This type of wear is manifested by a system of surface grooves and scratches, often called *scoring*. The abrasive wear condition in which the hard asperities of one surface wear away the mating surface is commonly called *two-body wear*, and the condition in which hard abrasive particles between the two surfaces cause the wear is called *three-body wear*.

An average abrasive wear depth d_{abr} may then be estimated as

$$d_{\text{abr}} = \frac{V_{\text{abr}}}{A_a} = \left(\frac{(\tan\,\theta)_m}{3\pi\sigma_{\text{yp}}}\right)\left(\frac{W}{A_a}\right) L_s \tag{33}$$

$$d_{\text{abr}} = k_{\text{abr}} p_m L_s \tag{34}$$

where W is total applied load, θ is the angle a typical conical asperity makes with respect to the direction of sliding, $(\tan\,\theta)_m$ is a weighted mean value for all asperities, L_s is a total distance of sliding, σ_{yp} is the uniaxial yield point strength for the softer material, V_{abr} is abrasive wear volume, $p_m = W/A_a$ is the mean nominal contact pressure between bearing surfaces, and $k_{\text{abr}} = (\tan\,\theta)_m/3\pi\sigma_{\text{yp}}$ is an abrasive wear coefficient that depends on the roughness characteristics of the surface and the yield strength (or hardness) of the softer material.

Comparing (33) for abrasive wear volume with (30) for adhesive wear volume, we note that they are formally the same except the constant $k/3$ in the adhesive wear equation is replaced by $(\tan\,\theta)_m/\pi$ in the abrasive wear equation. Typical values of the wear constant $3(\tan\,\theta)_m/\pi$ for several materials are shown in Table 8. As indicated in Table 8, experimental evidence shows that k_{abr} for three-body wear is typically about an order of magnitude smaller than for the two-body case, probably because the trapped particles tend to roll much of the time and cut only a small part of the time.

Table 7 Adhesive Wear Behavior of Various Pairs[a]

	Material Combination				
Description of Metal Pair	Al Disk	Steel Disk	Cu Disk	Ag Disk	Remarks
Soluble pairs with poor adhesive wear resistance	Be	Be	Be	Be	These pairs substantiate the criteria of solubility and B subgroup metals
	Mg	—	Mg	Mg	
	Al	Al	Al	—	
	Si	Si8	Si	Si	
	Ca	—	Ca	—	
	Ti	Ti	Ti	—	
	Cr	Cr	—	—	
	—	Mn	—	—	
	Fe	Fe	—	—	
	Co	Co	Co	—	
	Ni	Ni	Ni	—	
	Cu	—	Cu	—	
	—	Zn	Zn	—	
	Zr	Zr	Zr	Zr	
	Nb	Nb	Nb	—	
	Mo	Mo	Mo	—	
	Rh	Rh	Rh	—	
	—	Pd	—	—	
	Ag	—	Ag	—	
	—	—	Cd	Cd	
	—	—	In	In	
	Sn	—	Sn	—	
	Ce	Ce	Ce	—	
	Ta	Ta	Ta	—	
	W	W	W	—	
	—	Ir	—	—	
	Pt	Pt	Pt	—	
	Au	Au	Au	Au	
	Th	Th	Th	Th	
	U	U	U	U	
Soluble pairs with fair or good adhesive wear resistance. (F) = Fair	—	Cu(F)	—		These pairs do not substantiate the stated criteria
	Zn(F)	—	—		
	—	—	Sb(F)		
Insoluble pairs, neither from the B subgroup, with poor adhewsive wear resistance		Li			These pairs substantiate the stated criteria
		Mg			
		Ca			
		Ba			
Insoluble pairs, one from the B subgroup, with fair or good adhesive wear resistance. (F) = Fair	—	C(F)	—	—	These pairs substantiate the stated criteria
	—	—	—	Ti(F)	
	—	—	Cr(F)	Cr(F)	
	—	—	—	Fe(F)	
	—	—	—	Co(F)	
	—	—	Ge(F)	—	
	—	Se(F)	Se(F)	—	
	—	—	—	Nb(F)	
	—	Ag	—	—	
	Cd	Cd	—	—	
	Ln	In	—	—	
	—	Sn(F)	—	—	
	—	Sb(F)	Sb	—	
	Te(F)	Te(F)	Te(F)	—	
	Ti	Ti	Ti	—	
	Pb(F)	Pb	Pb	—	
	Bi(F)	Bi	Bi(F)	—	
Insoluble pairs, one from the B subgroup, with poor adhesive wear resistance	C	—	C	C	These pairs do not substantiate the stated criteria
	—	—	—	Ni	
	Se	—	—	—	
	—	—	—	Mo	

[a] See pp. 34–35 of Ref. 72.

Table 8 Abrasive Wear Constant $3(\tan\theta)_m/\pi$ for Various Materials in Sliding Contact as Reported by Different Investigators

Materials	Wear Type	Particle Size (μm)	$3(\tan\theta)_m/\pi$
Many	Two body	—	180×10^{-3}
Many	Two body	110	150×10^{-3}
Many	Two body	40–150	120×10^{-3}
Steel	Two body	260	80×10^{-3}
Many	Two body	80	24×10^{-3}
Brass	Two Body	70	16×10^{-3}
Steel	Three body	150	6×10^{-3}
Steel	Three body	80	4.5×10^{-3}
Many	Three body	40	2×10^{-3}

Source: See p. 169 of Ref. 71. Reprinted with permission from John Wiley & Sons.

In selecting materials for abrasive wear resistance, it has been established that both hardness and modulus of elasticity are key properties. Increasing wear resistance is associated with higher hardness and lower modulus of elasticity since both the amount of elastic deformation and the amount of elastic energy that can be stored at the surface are increased by higher hardness and lower modulus of elasticity.

Table 9 tabulates several materials in order of descending values of (hardness)/(modulus of elasticity). Well-controlled experimental data are not yet available, but general experience would provide an ordering of materials for decreasing

Table 9 Values of (Hardness/Modulus of Elasticity) for Various Materials

Material	Condition	BHN[a] ($E \times 10^{-6}$) (in mixed units)
Alundum (Al_2O_3)	Bonded	143
Chrome plate	Bright	83
Gray ion	Hard	33
Tungsten carbide	9% Co	22
Steel	Hard	21
Titanium	Hard	17
Aluminum alloy	Hard	11
Gray iron	As cast	10
Structural steel	Soft	5
Malleable iron	Soft	5
Wrought iron	Soft	3.5
Chromium metal	As cast	3.5
Copper	Soft	2.5
Silver	Pure	2.3
Aluminum	Pure	2.0
Lead	Pure	2.0
Tin	Pure	0.7

[a] Brinell hardness number.

Source: Reprinted from Ref. 59, Copyright 1957, with permission from Elsevier Science.

wear resistance compatible with the array of Table 9. When the conditions for adhesive or abrasive wear exist together with conditions that lead to corrosion, the two processes persist together and often interact synergistically. If the corrosion product is hard and abrasive, dislodged corrosion particles trapped between contacting surfaces will accelerate the abrasive wear process. In turn, the wear process may remove the ''protective'' surface layer of corrosion product to bare new metal to the corrosive atmosphere, thereby accelerating the corrosion process. Thus, the corrosion wear process may be self-accelerating and may lead to high rates of wear.

On the other hand, some corrosion products, for example, metallic phosphates, sulfides, and chlorides, form as soft lubricative films that actually improve the wear rate markedly, especially if adhesive wear is the dominant phenomenon.

Three major wear control methods have been defined, as follows (see p. 36 of Ref. 72): *principle of protective layers,* including protection by lubricant, surface film, paint, plating, phosphate, chemical, flame-sprayed, or other types of interfacial layers; *principle of conversion,* in which wear is converted from destructive to permissible levels through better choice of metal pairs, hardness, surface finish, or contact pressure; and *principle of diversion,* in which the wear is diverted to an economical replaceable wear element that is periodically discarded and replaced as ''wear out'' occurs.

When two surfaces operate in rolling contact, the wear phenomenon is quite different from the wear of sliding surfaces just described, although the ''delamination'' theory[73] is very similar to the mechanism of wear between rolling surfaces in contact as described here. Rolling surfaces in contact result in Hertz contact stresses that produce maximum values of shear stress slightly below the surface. (See, for example, Ref. 74.) As the rolling contact zone moves past a given location on the surface, the subsurface peak shear stress cycles from zero to a maximum value and back to zero, thus producing a cyclic stress field. Such conditions may lead to fatigue failure by the initiation of a subsurface crack that propagates under repeated cyclic loading and that may ultimately propagate to the surface to spall out a macroscopic surface particle to form a wear pit. This action, called *surface fatigue wear,* is a common failure mode in antifriction bearings, gears, and cams, and all machine parts that involve rolling surfaces in contact.

8 CORROSION AND STRESS CORROSION

Corrosion may be defined as the undesired deterioration of a material through chemical or electrochemical interaction with the environment, or destruction of materials by means other than purely mechanical action. Failure by corrosion occurs when the corrosive action renders the corroded device incapable of performing its design function. Corrosion often interacts synergistically with another failure mode, such as wear or fatigue, to produce the even more serious combined failure modes, such as corrosion wear or corrosion fatigue. Failure by corrosion and protection against failure by corrosion has been estimated to cost in excess of $8 billion annually in the United States alone.[75]

The complexity of the corrosion process may be better appreciated by recognizing that many variables are involved, including environmental, electrochemical, and metallurgical aspects. For example, anodic reactions and rate of oxidation; cathodic reactions and rate of reduction; corrosion inhibition, polarization, or retardation; passivity phenomena; effect of oxidizers; effect of velocity; temperature; corrosive concentration; galvanic coupling; and metallurgical structure all influence the type and rate of the corrosion process.

Corrosion processes have been categorized in many different ways. One convenient classification divides corrosion phenomena into the following types[75,76]:

1. Direct chemical attack
2. Galvanic corrosion
3. Crevice corrosion
4. Pitting corrosion
5. Intergranular corrosion
6. Selective leaching
7. Erosion corrosion
8. Cavitation corrosion
9. Hydrogen damage
10. Biological corrosion
11. Stress corrosion cracking

Depending on the types of environment, loading, and mechanical function of the machine parts involved, any of the types of corrosion may combine their influence with other failure modes to produce premature failures. Of particular concern are interactions that lead to failure by corrosion wear, corrosion fatigue, fretting fatigue, and corrosion-induced fracture.

8.1 Types of Corrosion

Direct chemical attack is probably the most common type of corrosion. Under this type of corrosive attack the surface of the machine part exposed to the corrosive media is attacked more or less uniformly over its entire surface, resulting in a progressive deterioration and dimensional reduction of sound load-carrying net cross section. The rate of corrosion due to direct attack can usually be estimated from relatively simple laboratory tests in which small specimens of the selected material are exposed to a well-simulated actual environment, with frequent weight change and dimensional measurements carefully taken. The corrosion rate is usually expressed in mils per year (mpy) and may be calculated as[75]

$$R = \frac{534W}{\gamma A t} \tag{35}$$

where R is rate of corrosion penetration in mils (1 mil = 0.001 in.) per year (mpy), W is weight loss in milligrams, A is exposed area of the specimen in square inches, γ is density of the specimen in grams per cubic centimeter, and t is exposure time in hours. Use of this corrosion rate expression in predicting corrosion penetration in actual service is usually successful if the environment has been properly simulated in the laboratory. Corrosion rate data for many different combinations of materials and environments are available in the literature.[77–79] Figure 33 illustrates one presentation of such data.

Direct chemical attack may be reduced in severity or prevented by any one or a combination of several means, including selecting proper materials to suit the environment; using plating, flame spraying, cladding, hot dipping, vapor deposition, conversion coatings, and organic coatings or paint to protect the base material; changing the environment by using lower temperature or lower velocity, removing oxygen, changing corrosive concentration, or adding corrosion inhibitors; using cathodic protection in which electrons are supplied to the metal surface to be protected either by galvanic coupling to a sacrificial anode or by an external power supply; or adopting other suitable design modifications.

Galvanic corrosion is an accelerated electrochemical corrosion that occurs when two dissimilar metals in electrical contact are made part of a circuit completed by a connecting pool or film of electrolyte or corrosive medium. Under these circumstances, the potential difference between the dissimilar metals produces a current flow through the connecting electrolyte, which leads to corrosion,

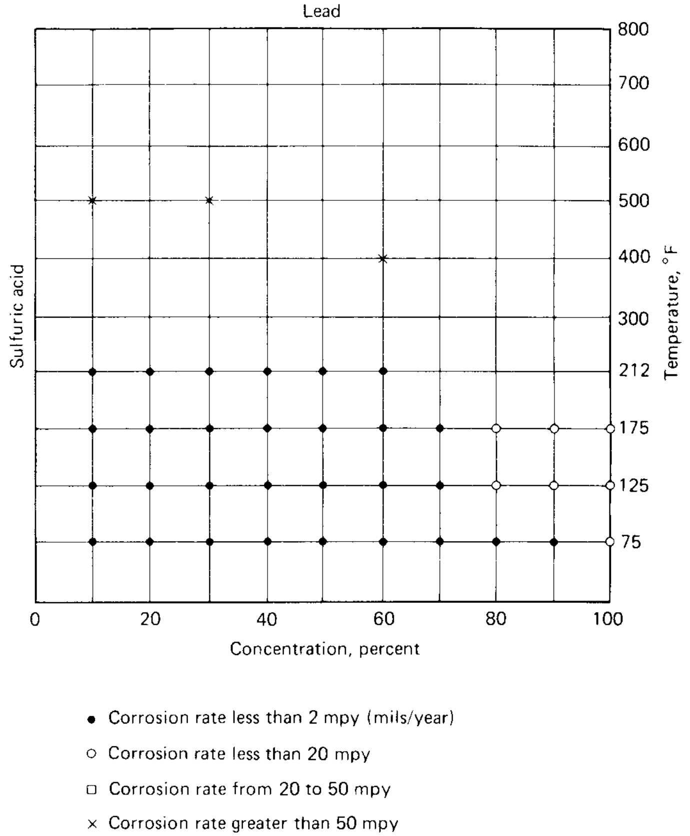


Fig. 33 Nelson's method for summarizing corrosion rate data for lead in sulfuric acid environment as a function of concentration and temperature. (After Ref. 77; reprinted with permission of McGraw-Hill Book Company)

concentrated primarily in the more anodic or less noble metal of the pair. This type of action is completely analogous to a simple battery cell. Current must flow to produce galvanic corrosion, and, in general, more current flow means more serious corrosion. The relative tendencies of various metals to form galvanic cells, and the probable direction of the galvanic action, are illustrated for several commercial metals and alloys in seawater in Table 10.[75,76]

Ideally, tests in the actual service environment should be conducted; but, if such data are unavailable, the data of Table 10 should give a good indication of possible galvanic action. The farther apart the two dissimilar metals are in the galvanic series, the more serious the galvanic corrosion problem may be. Material pairs within any bracketed group exhibit little or no galvanic action. It should be noted, however, that there are sometimes exceptions to the galvanic series of Table 10, so wherever possible corrosion tests should be performed with actual materials in the actual service environment.

The accelerated galvanic corrosion is usually most severe near the junction between the two metals, decreasing in severity at locations farther from the junction. The ratio of cathodic area to anodic area exposed to the electrolyte has a significant effect on corrosion rate. It is *desirable* to have a *small ratio* of cathode area to anode area. For this reason, if only *one* of two dissimilar metals in electrical contact is to be coated for corrosion protection, the more noble or more corrosion-resistant metal should be coated. Although this at first may seem the wrong metal to coat, the area effect, which produces anodic corrosion rate of 10^2–10^3 times cathodic corrosion rates for equal areas, provides the logic for this assertion.

Galvanic corrosion may be reduced in severity or prevented by one or a combination of several steps, including the selection of material pairs as close together as possible in the galvanic series, preferably in the same bracketed group; electrical insulation of one dissimilar metal from the other as completely as possible; maintaining as small a ratio of cathode area to anode area as possible; proper use and maintenance of coatings; the use of inhibitors to decrease the aggressiveness of the corroding medium; and the use of cathodic protection in which a third metal element anodic to both members of the operating pair is used as a sacrificial anode that may require periodic replacement.

Crevice corrosion is an accelerated corrosion process highly localized within crevices, cracks, and other small-volume regions of stagnant solution in contact with the corroding metal. For example, crevice corrosion may be expected in gasketed joints; clamped interfaces; lap joints; rolled joints; under bolt and rivet heads; and under foreign deposits of dirt, sand, scale, or corrosion product. Until recently, crevice corrosion was thought to result from differences in either oxygen concentration or metal ion concentration in the crevice compared to its surroundings. More recent studies seem to indicate, however, that the local oxidation and reduction reactions result in oxygen depletion in the stagnant crevice region, which leads to an excess positive charge in the crevice due to increased metal ion concentration. This, in turn, leads to a flow of chloride and hydrogen ions into the crevice, both of which accelerate the corrosion rate within the crevice. Such accelerated crevice corrosion is highly localized and often requires a lengthy incubation period of perhaps many months before it gets under way. Once started, the rate of corrosion accelerates to become a serious problem. To

Table 10 Galvanic Series of Several Commercial Metals and Alloys in Seawater

↑	Platinum
	Gold
Noble or cathodic (protected end)	Graphite
	Titanium
	Silver
	Chlorimet 3 (62 Ni, 18 Cr, 18 Mo) Hastelloy C (62 Ni, 17 C, 15 Mo)
	18-8 Mo stainless steel (passive) 18-8 stainless steel (passive) Chromium stainless steel 11–30% Cr (passive)
	Inconel (passive) (80 Ni, 13 Cr, 7 Fe Nickel (passive)
	Silver solder
	Monel (70 Ni, 30 Cu) Cupronickels (60-90 Cu, 40-10 Ni) Bronzes (Cu–Sn) Copper Brasses (Cu–Zn)
	Chlorimet 2 (66 Ni, 32 Mo, 1 Fe) Hastelloy B (60 Ni, 30 Mo, 6 Fe, 1 Mn)
	Inconel (active) Nickel (active)
	Tin
	Lead
	Lead–tin solders
	18-8 Mo stainless steel (active) 18-8 stainless steel (active)
	Ni-Resist (high Ni cast iron)
	Chromium stainless steel, 13% Cr (active)
	Cast iron Steel or iron
Active or anodic (corroded end)	2024 aluminum (4.5 Cu, 1.5 Mg, 0.6 Mn)
	Cadmium
	Commercially pure aluminum (1100)
	Zinc
↓	Magnesium and magnesium alloys

Source: Ref. 75. Reprinted with permission of McGraw-Hill Book Company.

be susceptible to crevice corrosion attack, the stagnant region must be wide enough to allow the liquid to enter but narrow enough to maintain stagnation. This usually implies cracks and crevices of a few thousandths to a few hundredths of an inch in width.

To reduce the severity of crevice corrosion, or prevent it, it is necessary to eliminate the cracks and crevices. This may involve caulking or seal welding existing lap joints; redesign to replace riveted or bolted joints by sound, welded joints; filtering foreign material from the working fluid; inspection and removal of corrosion deposits; or using nonabsorbent gasket materials.

Pitting corrosion is a very localized attack that leads to the development of an array of holes or pits that penetrate the metal. The pits may be widely scattered or so heavily concentrated that they simply appear as a rough surface. The mechanism of pit growth is virtually identical to that of crevice corrosion described, except that an existing crevice is not required to initiate pitting corrosion. The pit is probably initiated by a momentary attack due to a random variation in fluid concentration or a tiny surface scratch or defect. Some pits may become inactive because of a stray convective current, whereas others may grow large enough to provide a stagnant region of stable size, which then continues to grow over a long period of time at an accelerating rate. Pits usually grow in the direction of the gravity force field since the dense concentrated solution in a pit is required for it to grow actively. Most pits, therefore, grow downward from horizontal surfaces to ultimately perforate the wall. Fewer pits are formed on vertical walls, and very few pits grow upward from the bottom surface.

Measurement and assessment of pitting corrosion damage is difficult because of its highly local nature. Pit depth varies widely and, as in the case of fatigue damage, a statistical approach must be taken in which the probability of a pit of specified depth may be established in laboratory testing. Unfortunately, a significant size effect influences depth of pitting, and this must be taken into account when predicting service life of a machine part based on laboratory pitting corrosion data.

The control or prevention of pitting corrosion consists primarily of the wise selection of material to resist pitting or, since pitting is usually the result of stagnant conditions, imparting velocity to the fluid. Increasing its velocity may also decrease pitting corrosion attack.

Because of the atomic mismatch at the grain boundaries of polycrystalline metals, the stored strain energy is higher in the grain boundary regions than in the grains themselves. These high-energy grain boundaries are more chemically reactive than the grains. Under certain conditions depletion or enrichment of an alloying element or impurity concentration at the grain boundaries may locally change the composition of a corrosion-resistant metal, making it susceptible to corrosive attack. Localized attack of this vulnerable region near the grain boundaries is called intergranular corrosion. In particular, the austenitic stainless steels are vulnerable to intergranular corrosion if *sensitized* by heating into the temperature range from 950 to 1450°F, which causes depletion of the chromium near the grain boundaries as chromium carbide is precipitated at the boundaries. The chromium-poor regions then corrode because of local galvanic cell action, and the grains literally fall out of the matrix. A special case of intergranular corrosion, called "weld decay," is generated in the portion of the weld heat-affected zone, which is heated into the sensitizing temperature range.

To minimize the susceptibility of austenitic stainless steels to intergranular corrosion, the carbon content may be lowered to below 0.03%, stabilizers may be added to prevent depletion of the chromium near the grain boundaries, or a high-temperature solution heat treatment, called quench-annealing, may be employed to produce a more homogeneous alloy.

Other alloys susceptible to intergranular corrosion include certain aluminum alloys, magnesium alloys, copper-based alloys, and die-cast zinc alloys in unfavorable environments.

The corrosion phenomenon in which one element of a solid alloy is removed is termed selective leaching. Although the selective leaching process may occur in any of several alloy systems, the more common examples are *dezincification* of brass alloys and *graphitization* of gray cast iron. Dezincification may occur as either a highly local "plug-type" or a broadly distributed layer-type attack. In either case, the dezincified region is porous, brittle, and weak. Dezincification may be minimized by adding inhibitors such as arsenic, antimony, or phosphorous to the alloy; by lowering oxygen in the environment; or by using cathodic protection.

In the case of graphitization of gray cast iron, the environment selectively leaches the iron matrix to leave the graphite network intact to form an active galvanic cell. Corrosion then proceeds to destroy the machine part. Use of other alloys, such as nodular or malleable cast iron, mitigates the problem because there is no graphite network in these alloys to support the corrosion residue. Other alloy systems in adverse environments that may experience selective leaching include aluminum bronzes, silicon bronzes, and cobalt alloys.

Erosion corrosion is an accelerated, direct chemical attack of a metal surface due to the action of a moving corrosive medium. Because of the abrasive wear action of the moving fluid, the formation of a protective layer of corrosion product is inhibited or prevented, and the corroding medium has direct access to bare, unprotected metal. Erosion corrosion is usually characterized by a pattern of grooves or peaks and valleys generated by the flow pattern of the corrosive medium. Most alloys are susceptible to erosion corrosion, and many different types of corrosive media may induce erosion corrosion, including flowing gases, liquids, and solid aggregates. Erosion corrosion may become a problem in such machine parts as valves, pumps, blowers, turbine blades and nozzles, conveyors, and piping and ducting systems, especially in the regions of bends and elbows.

Erosion corrosion is influenced by the velocity of the flowing corrosive medium, turbulence of the flow, impingement characteristics, concentration of abrasive solids, and characteristics of the metal alloy surface exposed to the flow. Methods of minimizing or preventing erosion corrosion include reducing the velocity, eliminating or reducing turbulence, avoiding sudden changes in the direction of flow, eliminating direct impingement where possible, filtering out abrasive particles, using harder and more corrosion-resistant alloys, reducing the temperature, using appropriate surface coatings, and using cathodic protection techniques.

Cavitation often occurs in hydraulic systems, such as turbines, pumps, and piping, when pressure changes in a flowing liquid give rise to the formation and collapse of vapor bubbles at or near the containing metal surface. The impact associated with vapor bubble collapse may produce high-pressure shock waves that may plastically deform the metal locally or destroy any protective surface film of corrosion product and locally accelerate the corrosion process. Furthermore, the tiny depressions so formed act as a nucleus for subsequent vapor bubbles, which continue to form and collapse at the same site to produce deep pits and pockmarks by the combined action of mechanical deformation and accelerated chemical corrosion. This phenomenon is called cavitation corrosion. Cavitation corrosion may be reduced or prevented by eliminating the cavitation through appropriate design changes. Smoothing the surfaces, coating the walls, using corrosion-resistant materials, minimizing pressure differences in the cycle, and using cathodic protection are design changes that may be effective.

Hydrogen damage, although not considered to be a form of direct corrosion, is often induced by corrosion. Any damage caused in a metal by the presence of hydrogen or the interaction with hydrogen is called hydrogen damage. Hydrogen damage includes hydrogen blistering, hydrogen embrittlement, hydrogen attack, and decarburization.

Hydrogen blistering is caused by the diffusion of hydrogen atoms into a void within a metallic structure where they combined to form molecular hydrogen. The hydrogen pressure builds to a high level that, in some cases, causes blistering, yielding, and rupture. Hydrogen blistering may be minimized by using materials without voids, by using corrosion inhibitors, or by using hydrogen-impervious coatings.

Hydrogen embrittlement is also caused by the penetration of hydrogen into the metallic structure to form brittle hydrides and pin dislocation movement to reduce slip, but the exact mechanism is not yet fully understood. Hydrogen embrittlement is more serious at the higher-strength levels of susceptible alloys, which include most of the high-strength steels. Reduction and prevention of hydrogen embrittlement may be accomplished by "baking out" the hydrogen at relatively low temperatures for several hours, use of corrosion inhibitors, or use of less susceptible alloys.

Decarburization and hydrogen attack are both high-temperature phenomena. At high temperatures hydrogen removes carbon from an alloy, often reducing its tensile strength and increasing its creep rate. This carbon-removing process is called *decarburization*. It is also possible that the hydrogen may lead to the formation of methane in the metal voids, which may expand to form cracks, another form of hydrogen attack. Proper selection of alloys and coatings is helpful in prevention of these corrosion-related problems.

Biological corrosion is a corrosion process or processes that results from the activity of living organisms. These organisms may be microorganisms, such as aerobic or anaerobic bacteria, or they may be macroorganisms, such as fungi, mold, algae, or barnacles. The organisms may influence or produce corrosion by virtue of their processes of food ingestion and waste elimination. There are, for example, sulfate-reducing anaerobic bacteria, which produce iron sulfide when in contact with buried steel structures, and aerobic sulfur-oxidizing bacteria, which produce localized concentrations of sulfuric acid and serious corrosive attack on buried steel and concrete pipe lines. There are also iron bacteria, which ingest ferrous iron and precipitate ferrous hydroxide to produce local crevice corrosion attack. Other bacteria oxidize ammonia to nitric acid, which attacks most metals, and most bacteria produce carbon dioxide, which may form the corrosive agent carbonic acid. Fungi and mold assimilate organic matter and produce organic acids. Simply by their presence, fungi may provide the site for crevice corrosion attacks, as does the presence of attached barnacles and algae. Prevention or minimization of biological corrosion may be accomplished by altering the environment or by using proper coatings, corrosion inhibitors, bactericides or fungicides, or cathodic protection.

8.2 Stress Corrosion Cracking

Stress corrosion cracking is an extremely important failure mode because it occurs in a wide variety of different alloys. This type of failure results from a field of cracks produced in a metal alloy under the combined influence of tensile stress and a corrosive environment. The metal alloy is not attacked over most of its surface, but a system of intergranular or transgranular cracks propagates through the matrix over a period of time.

Stress levels that produce stress corrosion cracking are well below the yield strength of the material, and residual stresses as well as applied stresses may produce failure. The lower the stress level, the longer is the time required to produce cracking, and there appears to be a threshold stress level below which stress corrosion cracking does not occur.[75]

The chemical compositions of the environments that lead to stress corrosion cracking are highly specific and peculiar to the alloy system, and no general patterns have been observed. For example, austenitic stainless steels are susceptible to stress corrosion cracking in chloride environments but not in ammonia environments, whereas brasses are susceptible to stress corrosion cracking in ammonia environments but not in chloride environments. Thus, the "season cracking" of brass cartridge cases in the crimped zones was found to be stress corrosion cracking due to the ammonia resulting from decomposition of organic matter. Likewise, "caustic embrittlement" of steel boilers, which resulted in many explosive failures, was found to be stress corrosion cracking due to sodium hydroxide in the boiler water.

Stress corrosion cracking is influenced by stress level, alloy composition, type of environment, and temperature. Crack propagation seems to be intermittent, and the crack grows to a critical size, after which a sudden and catastrophic failure ensues in accordance with the laws of fracture mechanics. Stress corrosion crack growth in a statically loaded machine part takes place through the interaction of mechanical strains and chemical corrosion processes at the crack tip. The largest value of plane-strain stress intensity factor for which crack growth does not take place in a corrosive environment is designated K_{ISCC}. In many cases, corrosion fatigue behavior is also related to the magnitude of K_{ISCC}.[8]

Prevention of stress corrosion cracking may be attempted by lowering the stress below the critical threshold level, choice of a better alloy for the environment, changing the environment to eliminate the critical corrosive element, use of corrosion inhibitors, or use of cathodic protection. Before cathodic protection is implemented care must be taken to ensure that the phenomenon is indeed stress corrosion cracking because hydrogen embrittlement is accelerated by cathodic protection techniques.

9 FAILURE ANALYSIS AND RETROSPECTIVE DESIGN

In spite of all efforts to design and manufacture machines and structures to function properly without failure, failures do occur. Whether the failure consequences simply represent an annoying inconvenience or a catastrophic loss of life and property, it is the responsibility of the designer to glean all of the information possible from the failure event so that similar events can be avoided in the future. Effective assessment of service failures usually requires the intense interactive scrutiny of a team of specialists, including at least a mechanical designer and a materials engineer trained in failure analysis techniques. The team might often include a manufacturing engineer and a field service engineer as well. The mission of the failure analysis team is to discover the initiating cause of failure, identify the best solution, and redesign the product to prevent future failures. Although the results of failure analysis investigations may often be closely related to product liability litigation, the legal issues will not be addressed in this discussion.

Techniques utilized in the failure analysis effort include the inspection and documentation of the event through direct examination, photographs and eyewitness reports; preservation of all parts, especially failed parts; and pertinent calculations, analyses, and examinations that may help establish and validate the cause of failure. The materials engineer may utilize macroscopic examination, low-power magnification, microscopic examination, transmission or scanning electron microscopic techniques, energy-dispersive X-ray techniques, hardness tests, spectrographic analysis, metallographic examination, or other techniques of determining the failure type, failure location, material abnormalities, and potential causes of failure. The designer may perform stress and deflection analyses, examine geometry, assess service loading and environmental influences, reexamine the kinematics and dynamics of the application, and attempt to reconstruct the failure scenario. Other team members may examine the quality of manufacture, the quality of maintenance, the possibility of unusual or unconventional usage by the operator, or other factors that may have played a role in the service failure. Piecing all of this information together, it is the objective of the failure analysis team to identify as accurately as possible the probable cause of failure.

As undesirable as service failures may be, the results of a well-executed failure analysis may be transformed directly into improved product reliability by a designer who capitalizes on service failure data and failure analysis results. These techniques of retrospective design have become important working tools of the profession and are likely to continue to grow in importance.

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CHAPTER 26

FAILURE ANALYSIS OF PLASTICS

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1 INTRODUCTION

Part failure is generally related to one of four key factors: material selection, design, process, and service conditions.

1.1 Material Selection

Failures arising from hasty material selection are not uncommon in plastics or any other industry. In an application that demands high-impact resistance, a high-impact material must be specified. If the material is to be used outdoors for a long period, an ultraviolet resistant (UV) material must be specified. For proper material selection, careful planning, a thorough understanding of plastic materials, and reasonable prototype testing are required. The material selection should not be solely based on cost. A systematic approach to the material selection process is necessary to select the best material for any application. The proper material selection technique involves carefully defining the application requirement in terms of mechanical, thermal, environmental, electrical, and chemical properties. In many instances, it makes sense to design a thinner wall part taking advantage of the stiffness-to-weight ratio offered by higher-priced, fast cycling engineering materials. Many companies, including material suppliers, have developed software to assist in material selection simply by selecting application requirement in order of importance.

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1.2 Design

Proper material selection alone will not prevent a product from failing. While designing a plastic product, the designer must use the basic rules and guidelines provided by the material supplier for designing a particular part in that material. One must remember that with the exception of a few basic rules in designing plastic parts, the design criteria changes from material to material as well as from application to application. Today, design-related failures are by far the most common type of failure. Design guides for proper plastic part design are readily available from material suppliers. Table 1 shows a typical part design checklist.

1.3 Process

After proper material selection and design, the responsibility shifts from the designer to the plastic processor. The most innovative design and a very careful material selection cannot make up for poor processing practices. Molded-in stresses, voids, weak weld lines, and moisture in the material are some of the most common causes for premature product failures.

The latest advancement in process control technology allows the processors to not only control the process with a high degree of reliability but also helps in record keeping should a product fail at a later date. Such records of processing parameters are invaluable to a person conducting failure analysis. Any assembly or secondary operation on processed part must be evaluated carefully to avoid premature failures. Failures arising from stress cracking around metal inserts, drilled holes, and welded joints are quite common.

1.4 Service Conditions

In spite of the built-in safety factor warning labels, and user's instructions, failures arising from service conditions is quite common in the plastics industry. Five categories of unintentional service conditions are as follows.

1. Reasonable misuse
2. Use of product beyond its intended lifetime
3. Failure of product due to unstable service conditions
4. Failure due to service condition beyond reasonable misuse
5. Simultaneous application of two stresses operating synergistically

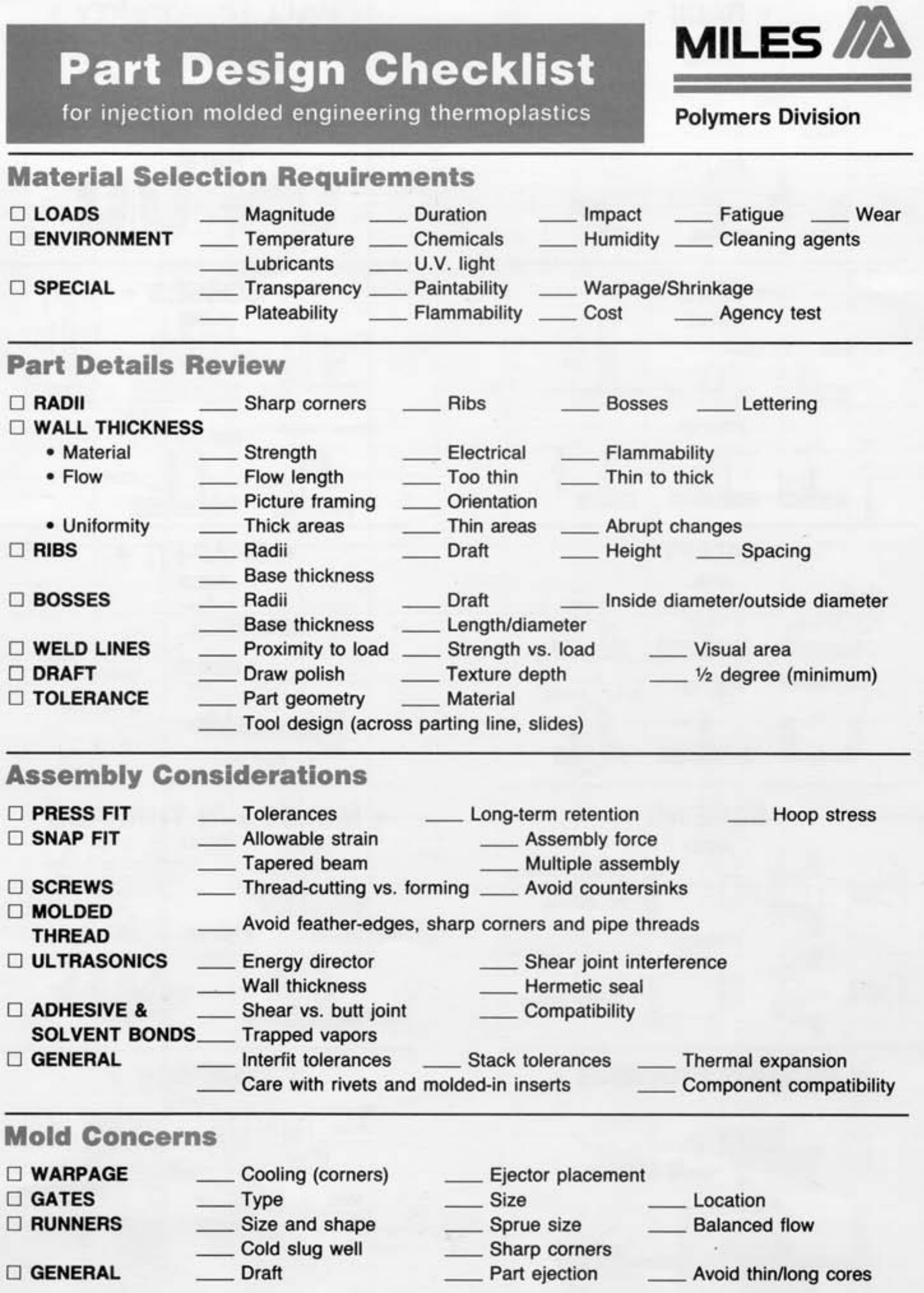
Most of the stresses imposed on plastics products in service can be grouped under the headings of thermal, chemical, physical, biological, mechanical, and electrical.

2 TYPES OF FAILURES

2.1 Mechanical Failure

Mechanical failure arises from the applied external forces that, when they exceed the yield strength of the material, cause the product to deform, crack, or break into pieces. The force may have been applied in tension, compression, and impact for a short or a long period of time at varying temperatures and humidity conditions.

Table 1 Part Design Checklist



Part Design Checklist

for injection molded engineering thermoplastics

MILES

Polymers Division

Material Selection Requirements

☐ **LOADS** ____ Magnitude ____ Duration ____ Impact ____ Fatigue ____ Wear
☐ **ENVIRONMENT** ____ Temperature ____ Chemicals ____ Humidity ____ Cleaning agents
____ Lubricants ____ U.V. light
☐ **SPECIAL** ____ Transparency ____ Paintability ____ Warpage/Shrinkage
____ Plateability ____ Flammability ____ Cost ____ Agency test

Part Details Review

☐ **RADII** ____ Sharp corners ____ Ribs ____ Bosses ____ Lettering
☐ **WALL THICKNESS**
- Material ____ Strength ____ Electrical ____ Flammability
- Flow ____ Flow length ____ Too thin ____ Thin to thick
  ____ Picture framing ____ Orientation
- Uniformity ____ Thick areas ____ Thin areas ____ Abrupt changes

☐ **RIBS** ____ Radii ____ Draft ____ Height ____ Spacing
____ Base thickness
☐ **BOSSES** ____ Radii ____ Draft ____ Inside diameter/outside diameter
____ Base thickness ____ Length/diameter
☐ **WELD LINES** ____ Proximity to load ____ Strength vs. load ____ Visual area
☐ **DRAFT** ____ Draw polish ____ Texture depth ____ ½ degree (minimum)
☐ **TOLERANCE** ____ Part geometry ____ Material
____ Tool design (across parting line, slides)

Assembly Considerations

☐ **PRESS FIT** ____ Tolerances ____ Long-term retention ____ Hoop stress
☐ **SNAP FIT** ____ Allowable strain ____ Assembly force
____ Tapered beam ____ Multiple assembly
☐ **SCREWS** ____ Thread-cutting vs. forming ____ Avoid countersinks
☐ **MOLDED THREAD** ____ Avoid feather-edges, sharp corners and pipe threads
☐ **ULTRASONICS** ____ Energy director ____ Shear joint interference
____ Wall thickness ____ Hermetic seal
☐ **ADHESIVE & SOLVENT BONDS** ____ Shear vs. butt joint ____ Compatibility
____ Trapped vapors
☐ **GENERAL** ____ Interfit tolerances ____ Stack tolerances ____ Thermal expansion
____ Care with rivets and molded-in inserts ____ Component compatibility

Mold Concerns

☐ **WARPAGE** ____ Cooling (corners) ____ Ejector placement
☐ **GATES** ____ Type ____ Size ____ Location
☐ **RUNNERS** ____ Size and shape ____ Sprue size ____ Balanced flow
____ Cold slug well ____ Sharp corners
☐ **GENERAL** ____ Draft ____ Part ejection ____ Avoid thin/long cores

2.2 Thermal Failure

Thermal failures occur from exposing products to an extremely hot or extremely cold environment. At abnormally high temperatures the product may warp, twist, melt, or even burn. Plastics tend to get brittle at low temperatures. Even the slightest amount of load may cause the product to crack or even shatter.

2.3 Chemical Failure

Very few plastics are totally impervious to all chemicals. Failure occurring from exposing the products to certain chemicals is quite common. Residual or molded-in stress, high temperatures, and external loading tend to aggravate the problem.

2.4 Environmental Failure

Plastics exposed to outdoor environments are susceptible to many types of detrimental factors. Ultraviolet rays, humidity, microorganisms, ozone, heat, and pollution are major environmental factors that seriously affect plastics. The effect can be anywhere from a mere loss of color, slight crazing and cracking to a complete breakdown of the polymer structure.

3 ANALYZING FAILURES

The first step in analyzing any type of failure is to determine the cause of the failure. Before proceeding with any elaborate tests, some basic information regarding the product must be gathered. If the product is returned from the field, have the district manager or consumer give you basic information, such as the date of purchase, date of installation, date when the first failure encountered, geographic location, types of chemicals used with or around the product, and whether the product was used indoors or outdoors. All this information is very vital if one is to analyze the defective product proficiently. For example, if the report from the field along with the defective product indicates a certain type of chemical was used with the product, one can easily check the chemical compatibility of the product or go one step further and simulate the actual-use condition using that same chemical. Record keeping also simplifies the task of failure analysis. A simple date code or cavity identification number will certainly enhance the traceability. Many types and styles of checklists to help analyze the failures have been developed. Seven basic methods are employed to analyze product failure: (1) visual examination, (2) identification analysis, (3) stress analysis, (4) microtoming, (5) mechanical testing, (6) thermal analysis, and (7) nondestructive testing (NDT) techniques. By zeroing in on the type of failure, one can easily select the appropriate method of failure analysis.

3.1 Visual Examination

A careful visual examination of the returned part can reveal many things. Excessive splay marks indicate that the materials were not adequately dried before processing. The failure to remove moisture from hydroscopic materials can lower the overall physical properties of the molded article and in some cases even cause them to become brittle. The presence of foreign material and other contaminants is also detrimental and could have caused the part to fail. Burn marks on molded articles are easy to detect. They are usually brown streaks and black spots. These marks indicate the possibility of material degradation during processing, causing the breakdown of molecular structure leading to overall reduction in the physical properties. Sink marks and weak weld lines, readily visible on molded parts, represent poor processing practices and may contribute to part failure.

A careful visual examination will also reveal the extent of consumer abuse. The presence of unusual chemicals, grease, pipe dope, and other substances may give some clues. Heavy marks and gouges could be the sign of excessively applied external force.

The defective part should also be cut in half using a sharp saw blade. The object here is to look for voids caused by trapped gas and excessive shrinkage, especially in thick sections during molding. A reduction in wall thickness caused by such voids could be less than adequate for supporting compressive or tensile force or withstanding impact load and may cause part to fail. Lastly, if the product has failed because of exposure to UV rays and other environmental factors, a slight chalking, microscopic cracks, large readily visible cracks, or loss of color will be evident.

3.2 Identification Analysis

One of the main reasons for product failure is simply use of the wrong material. When a defective product is returned from the field, material identification tests must be carried out to verify that the material used in the defective product is, in fact, the material specified on the product drawing. Chapter 21 discusses simple material identification techniques. However, identifying the type of material is simply not enough. Since all plastic materials are supplied in a variety of grades with a broad range of properties, the grade of material must also be determined. A simple technique such as the melt index test can be carried out to confirm the grade of a particular type of material. The percentage of regrind material mixed with virgin material has a significant effect on the physical properties. Generally, the higher the level of regrind material mixed with virgin, the lower the physical properties. If during processing higher than recommended temperature and long residence time are used, chances are that the material is degraded. This degraded material, when reground and mixed with virgin material, can cause a significant reduction in overall properties. Unfortunately, the percentage of regrind used with virgin is almost impossible to determine by performing tests on the molded parts. However, a correlation between the melt index value and the part failure rate can be established by conducting a series of tests to determine the minimum or maximum acceptable melt index value.

Part failures due to impurities and contamination of virgin material are quite common. Material contamination usually occurs during processing. A variety of purging materials are used to purge the previous material from the extruder barrel before using the new material. Not all of these purging materials are compatible. Such incompatibility can cause the loss of properties, brittleness, and delamination. In the vinyl compounding operation, failure to add key ingredients, such as an impact modifier, can result in premature part failure. Simple laboratory techniques cannot identify such impurities, contamination, or the absence of a key ingredient. More sophisticated techniques; such as Fourier transform infrared

(FT-IR) analysis and gel permeation chromatography (GPC) must be employed. These methods can not only positively identify the basic material but also point out the type and level of impurities in most cases.

3.3 Stress Analysis

Once the part failure resulting from poor molding practices or improper material usage through visual examination and material identification is ruled out, the next logical step is to carry out an experimental stress analysis. Experimental stress analysis is one of the most versatile methods for analyzing parts for possible failure. The part can be externally stressed or can have residual or molded-in stresses. External stresses or molded-in stresses or a combination of both can cause a part to fail prematurely. Stress analysis is an important part of failure identification. Detection of residual stresses has a different meaning than evaluation of stresses due to applied forces. It is possible of course to see failure due to poor design or underestimating of forces. These failures are usually detected in proof testing or in early production. Residual stresses are altogether different: A molding process can generate residual stress just about anywhere, anytime. Here, ongoing photoelastic inspection can prove extremely helpful, allowing detection of defective molded parts or identification of failures in clear plastic products. Experimental stress analysis can be conducted to determine the actual levels of stress in the part. Five basic methods are used to conduct stress analysis: photoelastic method, brittle-coating method, strain gauge method, chemical method, and heat reversion technique.

1. *Photoelastic Method.* The photoelastic method for experimental stress analysis is quite popular among design engineers and has proved to be an extremely versatile yet simple technique.

If the parts to be analyzed are made out of one of the transparent materials, stress analysis is simple. All transparent plastics, being birefringent, lend themselves to photoelastic stress analysis. The transparent part is placed between two polarizing mediums and viewed from the opposite side of the light source. The fringe patterns are observed without applying external stress. This allows the observer to study the molded-in or residual stresses in the part. High fringe order indicates the area of high-stress level whereas low fringe order represents an unstressed area. Also, close spacing of fringes represents a high-stress gradient. A uniform color indicates uniform stress in the part. Next, the part should be stressed by applying external force and simulating actual-use conditions. The areas of high-stress concentration can be easily pinpointed by observing changes in fringe patterns brought forth by external stress. Figure 1 illustrates a typical stress pattern in a part. This type of evaluation is useful as a regular part of product inspection for quality control of transparent parts for any manufacturer to maintain product quality and consistency and to prevent failures. The method often reveals problems associated with many other process control parameters such as temperature, material, fill rate, design, etc.

Another technique known as the photoelastic coating technique can be used to photoelastically stress-analyze opaque plastic parts. The part to be analyzed is coated with a photoelastic coating, service loads are applied to the part, and

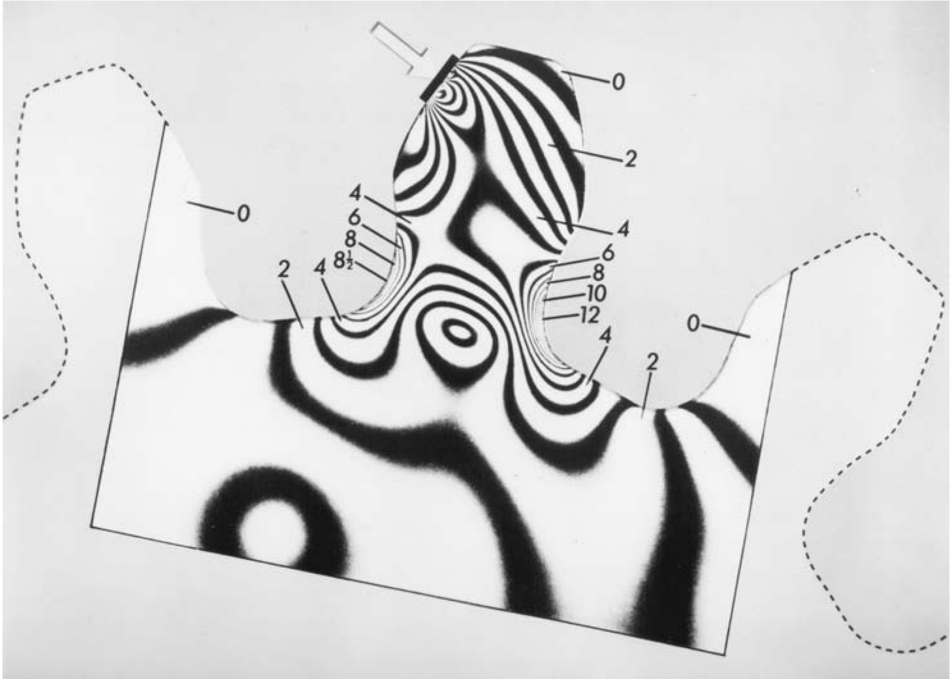


Fig. 1 Typical photoelastic stress pattern. (Courtesy Measurements Group, Inc.)

coating is illuminated by polarized light from the reflection polariscope. Molded-in or residual stresses cannot be observed with this technique. However, the same part can be fabricated using one of the transparent plastic materials. In summary, photoelastic techniques can be used successfully for failure analysis of a defective product.

2. *Brittle-Coating Method.* The brittle-coating method is yet another technique of conveniently measuring the localized stresses in a part. Brittle coatings are specially prepared lacquers that are usually applied by spraying on the actual part. The part is subjected to stress after air-drying the coating. The location of maximum strain and the direction of the principal strain is indicated by the small cracks that appear on the surface of the part as a result of external loading. Thus, the technique offers valuable information regarding the overall picture of the stress distribution over the surface of the part. The data obtained from the brittle-coating method can be used to determine the exact areas for strain gauge location and orientation, allowing precise measurement of the strain magnitude at points of maximum interest. They are also useful for the determination of stresses at stress concentration points that are too small or inconveniently located for installation of strain gauges. The brittle-coating technique, however, is not suitable for detailed quantitative analysis such as photoelasticity. Sometimes it is necessary to apply an undercoating prior to the brittle coating to promote adhesion and to minimize compatibility problems.

3. *Strain Gauge Method.* The electrical resistance strain gauge method is the most popular and widely accepted method for strain measurements. The strain gauge consists of a grid of strain-sensitive metal foil bonded to a plastic backing material. When a conductor is subjected to a mechanical deformation, its electrical resistance changes proportionally. This principle is applied in the operation of a strain gauge. For strain measurements, the strain gauge is bonded to the surface of a part with a special adhesive and then connected electrically to a measuring instrument. When the test part is subjected to a load, the resulting strain produced on the surface of the part is transmitted to the foil grid. The strain in the grid causes a change in its length and cross section, and produces a change in the resistivity of the grid material. This change in grid resistance, which is proportional to the strain, is then measured with a strain gauge recording instrument. In using strain gauges for failure analysis, care must be taken to test the adhesives for compatibility with particular plastics to avoid stress-cracking problems.

Residual or molded-in stresses can be directly measured with strain gauges using the hole drilling method. This method involves measuring a stress at a particular location, drilling a hole through the part to relieve the frozen-in stresses, and then remeasuring the stress. The difference between the two measurements is calculated as residual stress.

4. *Chemical Method.* Most plastics, when exposed to certain chemicals while under stress, show stress cracking. This phenomenon is used in stress analysis of molded parts. The level of molded-in or residual stress in a part molded in ABS (acrylonitrile butadiene styrene) material can be determined by this method. The part is immersed in a mixture of glacial acetic acid and water for 2 min at 73°F and later inspected for cracks that occur where tensile stress at the surface is greater than the critical stress. The part may also be externally stressed to a predetermined level and sprayed on with the chemical to determine critical stresses. Stress cracking curves for many types of plastics have been developed by material suppliers. If a defective product returned from the field appears to have stress-cracked, similar tests should be carried out to determine molded-in stresses as well as the effect of external loading by simulating end-use conditions. Failures of such types are seen often in parts where metal inserts are molded-in or inserted after molding. Three other tests, stain resistance test, solvent stress-cracking resistance, and environmental stress-cracking resistance (ESCR) are also employed to analyze failed parts. The acetone immersion test, to determine the quality of rigid polyvinyl chloride (PVC) pipe and fittings as indicated by their reaction to immersion in anhydrous acetone, is very useful. An unfused PVC compound attacked by an anhydrous acetone causes the material to swell, flake, or completely disintegrate. A properly fused PVC compound is impervious to anhydrous acetone and only a minor swelling, if any, is observed. Defective PVC pipe or fittings returned from the field are subjected to this test for failure analysis.

3.4 Heat Reversion Technique (ASTM F1057)

All plastics manufacturing process introduce some degree of stress in the finished product. The stresses in molded parts are commonly referred to as molded-in (residual) stresses. By reversing the process, by reheating the molded or extruded product, the presence of stress can be determined. The test is conducted by simply placing the entire specimen or a portion of the specimen in thermostatically controlled, circulating air oven and subjecting it to a predetermined temperature for a specified time. The specimens are visually examined for a variety of attributes. The degree and severity of warpage, blistering, wall separation, fish-scaling, and distortion in the gate area of molded parts indicate stress level. Stresses and molecular orientation effects in the plastic material are relieved, and the plastic starts to revert to more stable form. The temperature at which this begins to occur is important. If changes start below the heat distortion temperature of the material, high levels of stress and flow orientation are indicated. The test has been significantly improved by new methods including the attachment of strain gauges to critical regions of the part to carefully monitor initial changes in the shape. ASTM F1057 describes the standard practice for estimating the quality of extruded PVC pipe by the heat reversion technique.

3.5 Microtoming

Microtoming is a technique of slicing an ultrathin section from a molded plastic part for microscopic examination. This technique has been used by biologists and metallurgists for years, but only in the last decade has this technique been used successfully as a valuable failure analysis tool.

Microtoming begins with the skillful slicing of an 8- to 10-μm-thick section from a part and mounting the slice on a transparent glass slide. The section is then examined under a light transmission microscope equipped with a polarizer for photoelastic analysis. A high-power (1000×) microscope that will permit photographic recording of the structure in color is preferred. By examining the microstructure of a material, much useful information can be derived. For example, microstructural examination of a finished part that is too brittle may show that the melt temperature was either nonuniform or too low. The presence of unmelted particles is usually evident in such cases. Another reason for frequent failures of the injection-molded part is failure to apply sufficient time and pressure to freeze the gates. This causes the parts to be underpacked, which creates center-wall shrinkage voids. Figure 2 illustrates such shrinkage voids. Voids tend to reduce the load-bearing capabilities and toughness of a part through the concentration of stress in a weak area.

Contamination, indicated by abnormality in the microstructure, almost always creates some problems. Contamination caused by the mixing of different polymers can be detected through such analysis by carefully studying the differences in polymer structures. Quite often, a poor pigment dispersion also causes parts to be brittle. This is readily observable through the microtoming technique. To achieve optimum properties, additives such as glass fibers and fillers must disperse properly. Microtoming a glass-fiber-reinforced plastic part reveals the degree of bonding of the glass fiber to the resin matrix as well as the dispersion and orientation of glass fibers. Molded-in stresses as well as stresses resulting from external loading are readily observed under cross-polarized light because of changes in birefringence when the molecular structure is strained. Microtoming technique can also be applied to check the integrity of spin and ultrasonic or vibration welds.

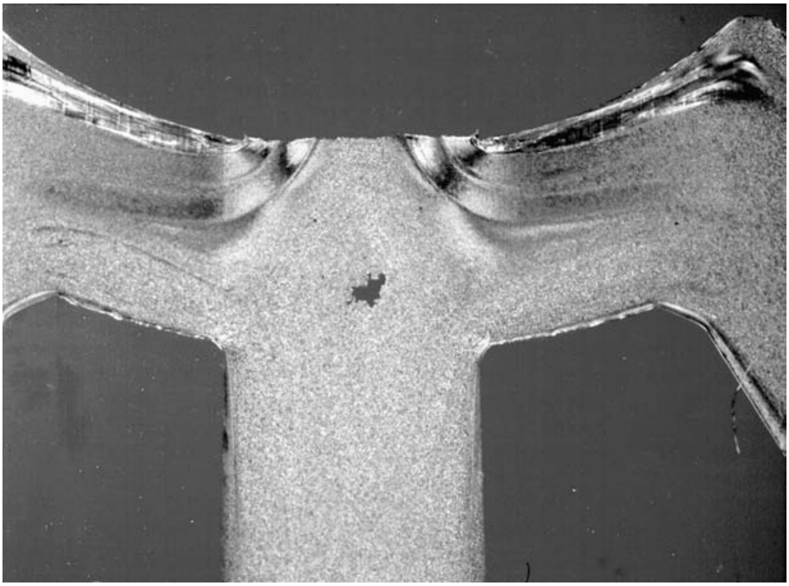


Fig. 2 Shrinkage voids created by insufficient time and pressure to freeze the gates during injection-molding process. (Courtesy BASF Corporation)

3.6 Mechanical Testing

Defective product returned from the field is often subjected to a variety of mechanical tests to determine the integrity of the product. Two basic methods are employed. One method involves conducting mechanical tests such as tensile, impact, or compression on the actual part or a small sample cut out from the part. The test results are then compared to the test results obtained from the retained samples. The second method requires grinding up the defective parts and either compression or injection-molding standard test bars and conducting mechanical tests. The test results are compared to the published data for the virgin material. The amount of material available for molding the test bars quite often precludes injection molding. The test data obtained from compression-molded samples are generally lower than injection-molded samples. Fatigue failure tests such as flexural fatigue or tensile fatigue can be employed to determine premature failure from cyclic loading.

3.7 Thermal Analysis

Thermal analysis techniques are used extensively in failure analysis. A detailed discussion on the use of thermogravimetric analysis (TGA), thermomechanical analysis (TMA), and differential scanning calorimetry (DSC) techniques is in a chapter on material characterization tests.

3.8 Nondestructive Testing Techniques

NDT techniques are useful in determining the flaws, discontinuities and joints.

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CHAPTER 27

FAILURE MODES: PERFORMANCE AND SERVICE REQUIREMENTS FOR CERAMICS

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1 OVERVIEW

A reliable design of components requires the knowledge of the possible failure modes under service conditions and knowledge of the material properties describing these failure modes. These material properties are characterized by specific material parameters, such as strength, fracture toughness, hardness, and relations between loading parameters and materials response, e.g., the stress–strain curve or the *S–N* curve for fatigue loading. A reliable design has to incorporate the uncertainties in the loading parameters and in the material response. Therefore, a correct description of the scatter of the material properties is an important part in any design procedure. Material properties usually are determined under simple loading conditions: uniaxial loading, homogeneous stress state, constant temperature, simple load–time variations, such as constant load, cyclic loading, or constant loading rate. The loading conditions of components are more complex: multiaxial and inhomogeneous stress state, complex

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load, and temperature variation. Therefore, methods to transform results from simple loading conditions to complex situations in components have to be developed and applied.

For metals, a good knowledge of failure modes, design rules following from these failure modes, and criteria for materials selection exists. Ceramics are brittle materials that fail without preceding plastic deformation. Only at high temperatures may creep deformation occur before fracture. The brittle failure mode requires specific design rules and criteria for material selection. Whereas in metals high local stresses lead to local plastic deformation only and not to failure of the component in ceramics, these high local stresses can lead to failure at relatively low global stresses. High local stresses can occur under different situations: sharp notches, thermal shock, contact loading. Tension stresses under these conditions are dangerous because of the lower tensile strength compared to compressive strength. From this behavior general design rules follow:

- Avoid sharp notches.
- Avoid surface damage, e.g., scratches.
- Avoid sudden changes in temperature.
- Avoid point or line loading of ceramic components.

The failure in ceramics starts from flaws introduced during fabrication or surface treatment. These flaws may be pores, inclusions, cracks, or surface scratches. Due to the scatter in the size of the flaws, also the strength shows a large scatter, which is much larger than in metals. This requires special statistical design criteria. This large scatter is associated with a large effect of the size of the component on the strength, which has to be included in the statistical design criteria.

Under mechanical or thermal loading, different failure modes are possible:

- Unstable fracture
- Subcritical extension of flaws under constant loading (static fatigue)
- Subcritical extension of flaws under cyclic loading (cyclic fatigue)
- Creep deformation and creep fracture at high temperatures
- Corrosion, especially oxidation
- Wear in components under sliding contact

The different failure modes are characterized by material properties and relations between the applied loading in terms of stresses or stress intensity factors and the materials response in terms of lifetime or plastic deformation. The material properties are:

- Tensile strength σ_c or, to be more specific, σ_{ct}
- Compressive strength σ_{cc}
- Fracture toughness K_{IC}
- Hardness H

The failure relations are:

- Relation between stress σ and lifetime t_f
- Relation between stress amplitude σ_a or stress range $\Delta\sigma = 2\sigma_a$ and number of cycles to failure N_f
- Relation between creep strain and time

The flaws leading to failure can be described as cracks. Therefore, the methods of fracture mechanics can be applied to describe the flaw extension behavior. The corresponding relations are:

- Relation between flaw size and strength
- Relation between crack growth rate and stress intensity factor for static fatigue
- Relation between crack extension for one load cycle and the range of the stress intensity factor for cyclic fatigue

Because of the scatter in strength and in the lifetime, the failure cannot be described by a unique strength or a fixed lifetime. For a given load, only the probability of failure can be specified. This failure probability depends not only on the maximum load in the component, it also depends on the size of the component and the stress distribution in the component.

2 FLAWS

Looking on a fracture surface of a ceramic, one very often can find the origin of the fracture. An example is shown in Fig. 1. It can distinguished between intrinsic and extrinsic flaws. Intrinsic flaws are generated during the fabrication process. They can be pores, shrinkage cracks, or areas of low density. Inclusions of other materials and specific grain configurations, especially large grains may

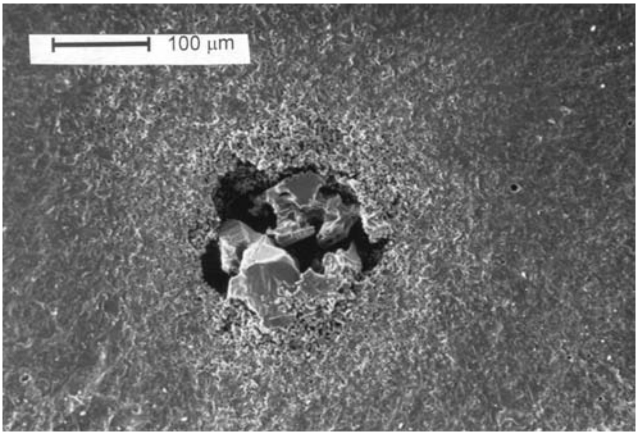

Fig. 1 Flaw origin in a silicon carbide.

lead to high microstructural residual stresses after cooling from the sintering temperature. These stresses may cause cracks during the application of a small mechanical load. Phase transformation during cooling or under an applied load, accompanied by a change in volume, e.g., in zirconia, is another cause of crack nucleation. All these flaws are primarily volume flaws that, however, may extend to the surface. Extrinsic flaws are surface flaws caused by surface treatment, such as machining or grinding. Oxidation at high temperature may introduce surface pits.

3 FRACTURE MECHANICS

The flaws that cause failure are described as cracks. Consequently, the methods of linear elastic fracture mechanics can be applied. The relation between the size of the flaw a and the strength σ_c is

$$\sigma_c = \frac{K_{\mathrm{IC}}}{\sqrt{a}Y} \tag{1}$$

where K_{IC} is the fracture toughness and Y a constant that depends on the shape of the crack. For a semicircular surface flaw there is $Y = 1.28$.

The fracture toughness is a material property. In Table 1 typical values of fracture toughness are listed. They range from less than 1 MPa$\sqrt{\mathrm{m}}$ for glass to 8 MPa$\sqrt{\mathrm{m}}$ for silicon nitride and 12 MPa$\sqrt{\mathrm{m}}$ for zirconia. For a given material the fracture toughness depends on the specific microstructure and density. Therefore, a large variation of K_{IC} is observed.

In Fig. 2 the strength σ_c according to Eq. 1 is plotted versus the crack size a for different values of K_{IC}. A flaw of a size of 100 μm leads to a strength of 391 MPa for a material with $K_{\mathrm{IC}} = 5$ MPa $\sqrt{\mathrm{m}}$ (e.g., alumina) and to a strength

Table 1 Fracture Toughness and Strength of Different Ceramics

	Fracture Toughness (MPa$\sqrt{\mathrm{m}}$)	Tensile Strength (MPa)	Compressive Strength (MPa)
AlN	3–4.5	300–500	
Al_2O_3	3–4.5	300–500	2500
Al_2TiO_5		10–50	
B_4C	3.5	300–500	2900
BeO		200–400	1500
$MoSi_2$		300	2500
SiC hot pressed	4.6	500–850	460
Si_3N_4 dense reaction bonded	4–8 1.5–2.8	500–1200 150–350	
SiO_2 fused quartz	0.8	50–100	1300 2000
TiB_2		300–400	1600
TiC		400	
ZrB_2		300	
ZrO_2	8–12	800–1000	2000
Glass	0.6–1.0		

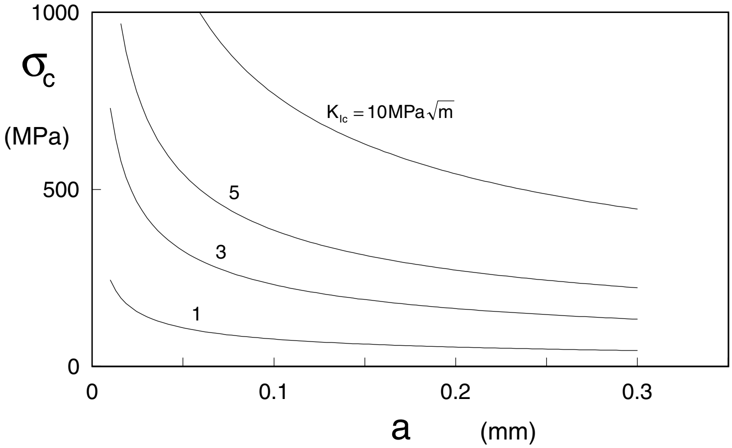

Fig. 2 Strength as a function of flaw size for materials with different fracture toughness.

of 781 MPa for a material with $K_{IC} = 10\ \text{MPa}\sqrt{\text{m}}$ (e.g., a zirconia). For a flaw size of 20 μm the corresponding values are 873 and 1747 MPa. In view of this large effect of the flaw size on the strength, a process technique that leads to a reduction of the processing flaws is desirable. Fracture may also be caused by surface flaws, introduced by grinding. Therefore, polishing the surface may also increase the strength.

The type of flaws also affects the strength. Applying Eq. 1 requires the existence of a crack. Three-dimensional flaws such as pores are described as spheres with a surrounding annular crack (Fig. 3). The stress intensity factor then depends on the ratio of the size of the crack *a* to the radius of the sphere *R*. The size of the crack may be in the order of the grain size.

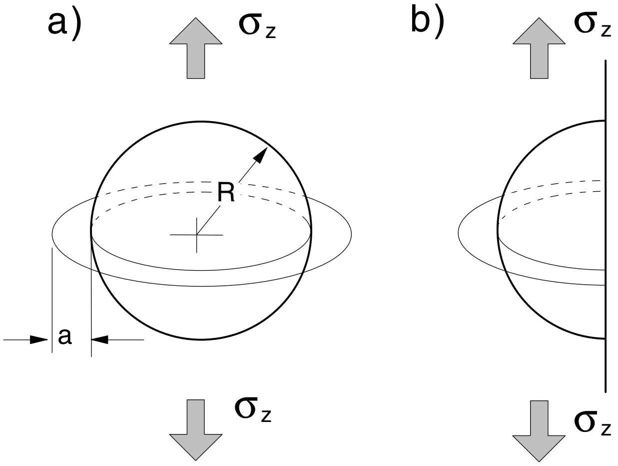

Fig. 3 Pores with annular crack.

4 STRENGTH

Equation 1 can be applied to predict the strength, if the crack size is known. In metals this relation is used, for instance, to predict the remaining strength of a component with a fatigue crack, whose size has been determined by a nondestructive testing method and is in the order of millimeters. In ceramic materials the flaws are small in the order of 10–100 μm and difficult to detect. Therefore Eq. 1 has not yet been used to predict the strength or to reject components after a nondestructive testing procedure. However, the relation shows the potential of a material. A material with a high fracture toughness and a low strength may have a potential for higher strength by a reduction in the flaw size.

In Fig. 4 the strength of SiC is plotted versus the crack size in a log-log plot. For cracks larger than about 100 μm a straight line with a slope of $-\frac{1}{2}$ is obtained according to Eq. 1. For smaller cracks a deviation with an upper limit is observed. The equivalent crack size is a length transformed to an internal crack in a plate. The reasons for the deviation from Eq. 1 for small flaw sizes shall not be discussed here.

Because of the large dependence of the strength on the flaw size for a given material, the strength exhibits a large variation, depending on the processing method of the material. Strength also can be varied for a given material by varying the grain size and the microstructure in multiphase materials because this may change the fracture toughness and the processing flaw size. The values given in Table 1 are typical values of dense materials. Generally, the tensile strengths of ceramics are lower than those of high-strength metals. Silicon nitride and some zirconia materials, however, have strength values of up to 1000 MPa.

5 DELAYED FAILURE

Under cyclic loading, but also under constant loading, ceramic materials may fail after some time. This is called static fatigue for constant loading and cyclic

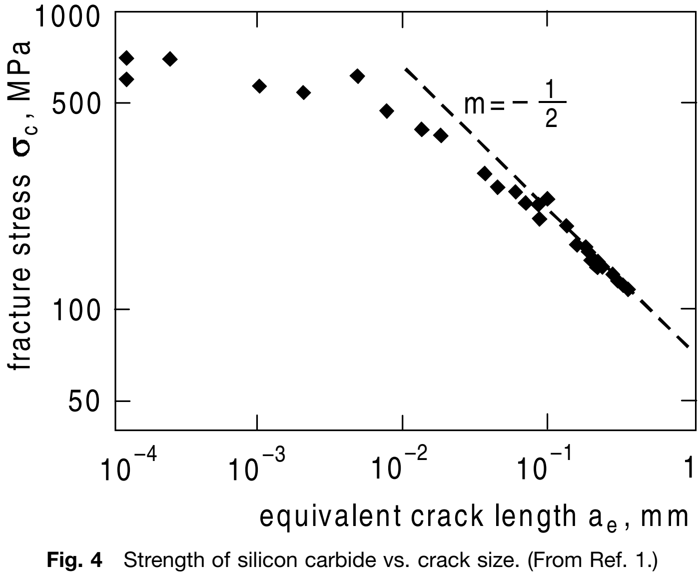

Fig. 4 Strength of silicon carbide vs. crack size. (From Ref. 1.)

fatigue for cyclic loading. This designation contrasts with that in metals, where the designation of fatigue is restricted to the damage under cyclic loading. The failure behavior is described by an *S–N* curve, where the stress is plotted versus the lifetime under static loading and the stress range $\Delta\sigma$ (twice the amplitude) versus the number of cycles to failure under cyclic loading. In a log-log plot straight lines are observed, which correspond to the relations:

$$t_f = \frac{A}{\sigma^n} \tag{2a}$$

$$N_f = \frac{A}{(\Delta\sigma)^n} \tag{2b}$$

The phenomenon of delayed failure is caused by subcritical extension of the flaws. For static loading the crack growth rate depends on the stress intensity factor K and for cyclic loading on the range of the stress intensity factor ΔK. In a large range of crack growth rate, the subcritical crack extension can be described by the relations of

$$\frac{da}{dt} = CK^n \tag{3a}$$

$$\frac{da}{dN} = C(\Delta K)^n \tag{3b}$$

where da/dN is the crack extension for one load cycle. Equation 3 can be obtained by integration of Eq. 4 from an initial crack size a_i to a critical crack size a_c. Because of the high values of n, the critical crack size can be set to infinity. The values of n in Eqs. 3 and 4 are the same for corresponding loading situations (static or cyclic). The parameters A, C, and n are different for static and cyclic loading, n_{cyclic} being always somewhat smaller than n_{static}. A and C for cyclic loading depend on the mean stress, which conveniently is expressed by the ratio of $R = \sigma_{\min}/\sigma_{\max} = K_{\min}/K_{\max}$.

The following relation was found:

$$\frac{da}{dN} = \frac{C}{(1 - R)^{n-p}} (\Delta K)^n \tag{4}$$

Integration leads to

$$N_f = C* \frac{(1 - R))^{n-p}}{(\Delta\sigma)^n} = C* \frac{1}{(\Delta\sigma)^p(\sigma_m + \Delta\sigma/2)^{n-p}} \tag{5}$$

Some values are given in Table 2. High values of n mean a large effect of the stress or the stress range on the lifetime.

Table 2 Some Values of *n* and *p*²

Material	*n*	*P*
ZrO_2 (Y-TZP)	19	3
Si_3N_4	29	1.3
SiC_w/Al_2O_3	10.2	4.8

Summary

- Delayed failure is described by a power law relation between crack growth rate and the stress intensity factor for constant loading and by a power law relation between crack extension for one load cycle and the range of the stress intensity factor for cyclic loading.

6 SCATTER OF STRENGTH AND LIFETIME

6.1 Scatter of Strength

Because of the statistical distribution of the flaw size, the strength is subjected to considerable scatter. As a consequence, the design of ceramic components cannot be based on an average strength and the application of a safety factor as it is usually done for metallic components. The scatter is associated with an effect of the size of a component and the stress distribution in the component on the strength. Therefore, a statistical treatment of the scatter and a design according to prescribed failure probability is necessary. In this section the method of applying Weibull statistics to uniaxial loading is described. The design applying multiaxial Weibull statistics will be presented in Section 7.

The scatter in the strength is described by the two-parameter Weibull distribution. The failure probability, i.e., the probability of failure occurring below the stress of $\sigma = \sigma_c$, is given by

$$F = 1 - \exp\left[-\left(\frac{\sigma_c}{\sigma_0}\right)^m\right] \tag{6}$$

The Weibull parameter m is called the Weibull modulus and a measure of the scatter. The Weibull parameter σ_0 is related to the strength. Whereas m is a material parameter, σ_0 depends on the material, but also on the size of the component and on the stress distribution in the component.

From Eq. 6 it follows that failure is possible also at very low stresses. Therefore, a three-parameter Weibull distribution with a lower limit σ_u is considered sometimes:

$$F = 1 - \exp\left[-\left(\frac{\sigma_c - \sigma_u}{\sigma_0}\right)^m\right] \qquad \sigma_c > \sigma_u \tag{7}$$

It is, however, not possible to determine σ_u with sufficient accuracy when applying a limited number of test specimens. Hence, the two-parameter distribution is used usually.

Failure can be caused by surface flaws or volume flaws. For a homogeneous stress distribution in a component and failure by volume flaws, Eq. 6 can be replaced by

$$F = 1 - \exp\left[-\frac{V}{V_0}\left(\frac{\sigma_c}{\sigma_V}\right)^m\right] \tag{8}$$

where V is the volume and V_0 is the unit volume, which is introduced for normalization. Now σ_V is a quantity independent of the size of the component. An inhomogeneous stress distribution in a component is described by the relation of

$$\sigma(x, y, z) = \sigma^* g(x, y, z) \tag{9}$$

where σ^* is a characteristic stress in the component and $g(x, y, z)$ a geometry function. The failure probability then is given by

$$F = 1 - \exp\left[-\frac{1}{V_0}\left(\frac{\sigma_c^*}{\sigma_V}\right)^m \int g^m \, dV\right] = 1 - \exp\left[-\frac{V_{\text{eff}}}{V_0}\left(\frac{\sigma_c^*}{\sigma_V}\right)^m\right] \tag{10}$$

where σ_c^* is the value of σ^* at fracture and

$$V_{\text{eff}} = \int g^m \, dV \tag{11}$$

is the effective volume.

For surface flaws the volume in Eqs. 8–11 has to be replaced by the surface area.

If Eq. 6 is used to calculate the failure probability for a component, $\sigma_{0,C}$ of the component can be calculated from $\sigma_{0,\text{SP}}$ of the specimen by

$$\sigma_{0,C} = \sigma_{0,\text{SP}}\left(\frac{V_{\text{eff, SP}}}{V_{\text{eff},C}}\right)^{1/m} \tag{12}$$

Experimental results are presented in a Weibull diagram, where ln ln $1/(1 - F)$ is plotted versus ln σ_c. To correlate the measured strength values with the failure probabilities, the strength values are ranked in increasing order and numbered from 1 to n. Then, the strength values σ_{ci} are related to the failure probability F_i according to

$$F_i = \frac{i - 0.5}{n} \tag{13}$$

The slope of the Weibull plot is m; σ_0 is the strength for $F = 0.632$. Examples are shown in Fig. 5.

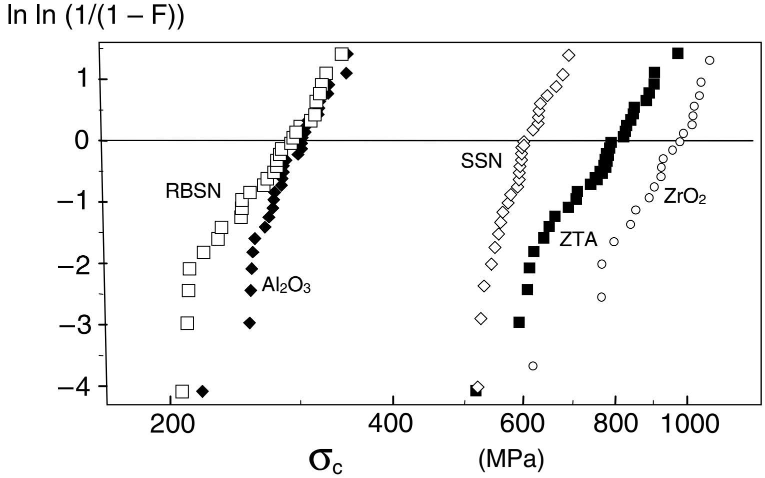

Fig. 5 Weibull plots of strength for several materials (RBSN: reaction bonded silicon nitride, SSN: sintered silicon nitride, ZTA: zirconia toughend alumina).

The average strength $\overline{\sigma_c}$ and the median strength for σ_{med} (σ_c for $F = 0.5$) are related to m and σ_0 by

$$\overline{\sigma_c} = \sigma_0\Gamma(1 + 1/m) \qquad \sigma_{\text{med}} = (0.693)^{1/m} \tag{14}$$

where Γ is the gamma function. In Table 3 the ratios $\overline{\sigma_c}/\sigma_0$ and $\sigma_{\text{med}}/\sigma_0$ are given for different values of m.

Equations 8 and 10 lead to a large effect of the size of the component on the strength, which depends on the Weibull modulus m. They also show a large effect of m on the failure probability. This effect is illustrated in Fig. 6 by the strength at different failure probabilities, Weibull parameters m, and different effective volumes. For a material with a mean strength of $\overline{\sigma_c} = 500$ MPa, which was determined with four-point specimens having an inner span $L = 20$ mm, width $W = 4.5$ mm, and height $H = 3.5$ mm. The effective volume of this specimen is

$$V_{\text{eff}} = \frac{LWH}{2(m + 1)} \tag{15}$$

In Table 4 the parameter σ_0 and the effective volume are given for different failure probabilities and different values of m.

Table 3 Ratios $\overline{\sigma_c}/\sigma_0$ and $\sigma_{\text{med}}/\sigma_0$

	$m = 5$	$m = 10$	$m = 20$	$m = 30$	$m = 50$
$\overline{\sigma_c}/\sigma_0$	0.918	0.951	0.974	0.982	0.989
$\sigma_{\text{med}}/\sigma_0$	0.929	0.964	0.982	0.988	0.993

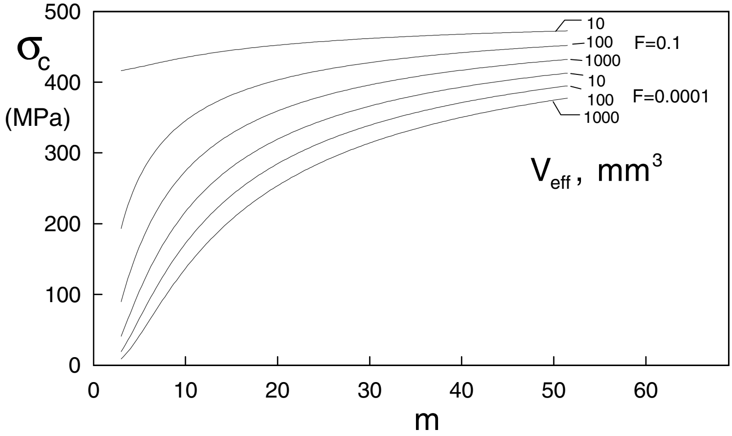

Fig. 6 Effect of Weibull modulus *m* and effective volume on the strength for two different failure probabilities for materials with the same average strength of 500 MPa.

6.2 Scatter of Lifetime

The scatter of the lifetime under constant or cyclic loading is much larger than that of the strength and can also described by a Weibull distribution. For static fatigue the failure probability is

$$F = 1 - \exp\left[-\left(\frac{t_f}{t_0}\right)^{m^*}\right] \tag{16a}$$

and for cyclic fatigue

$$F = 1 - \exp\left[-\left(\frac{N_f}{N_0}\right)^{m^*}\right] \tag{16b}$$

If the strength and lifetime are caused by the same flaws, then the Weibull

Table 4 Strength at Different Failure Probabilities and Effective Volumes (Materials with the Same Average Strength in Bending Tests)

m	V_{eff} (mm^3)	σ_0	F = 0.0001	F = 0.001	F = 0.01	F = 0.1	F = 0.5
5	10	661	105	166	264	4222	615
	100	417	66	105	168	266	388
	1000	263	42	66	195	168	245
10	10	545	217	273	435	435	526
	100	433	172	217	346	346	417
	1000	349	137	172	275	275	332
20	10	506	319	358	402	452	496
	100	451	284	319	358	403	442
	1000	402	253	284	319	359	394
50	10	495	411	431	451	473	491
	100	473	393	412	431	452	470
	1000	452	376	394	412	432	449

parameters m^*, t_0, and N_0 can be related to the Weibull parameters m and σ_0 of the strength:

$$m^* = \frac{m}{n - 2} \tag{17}$$

$$t_0 = \frac{B\sigma_0^{n-2}}{\sigma^n} \tag{18a}$$

$$N_0 = \frac{B\sigma_0^{n-2}}{(\Delta\sigma)^n} \tag{18b}$$

where n is the crack growth exponent for static and cyclic crack extension, respectively; B is given by

$$B = \frac{2}{(n - 2)CY^2K_{\mathrm{IC}}^{n-2}} \tag{19}$$

with C from Eq. (4).

In Eq. 18 $\sigma_{0,C}$ of the component has to be used. If $\sigma_{0,\mathrm{SP}}$ as obtained from the specimens is known, the parameters t_0 and N_0 in Eqs. 16 are

$$t_{0,C} = \frac{B\sigma_{0,\mathrm{SP}}^{n-2}}{\sigma^n}\left(\frac{V_{\mathrm{eff,\,SP}}}{V_{\mathrm{eff},\,C}}\right)^{(n-2)/m} \tag{20a}$$

$$N_{0,C} = \frac{B\sigma_{0,\mathrm{SP}}^{n-2}}{(\Delta\sigma)^n}\left(\frac{V_{\mathrm{eff,\,SP}}}{V_{\mathrm{eff},\,C}}\right)^{(n-2)/m} \tag{20b}$$

Summary

- The scatter of the strength is described by the two-parameter Weibull distribution with the Weibull modulus m.
- The average strength and the strength at given failure probability depend on the size of the component and the stress distribution in the component.
- The lifetime is described by a Weibull distribution; the parameters can be obtained from the Weibull parameters of the strength, if strength and lifetime are caused by the same flaws.

7 DESIGN APPLYING MULTIAXIAL WEIBULL STATISTICS

7.1 Strength Under Compression Loading

The strength of ceramic materials under compressive loading is much higher than under tension loading. Usually compression tests are performed applying cylindrical specimens. The cylinders are loaded with parallel stamps. Due to the different lateral expansion of the specimen and the stamp under compressive loading, radial stresses occur in the loaded specimen, which may lead to premature failure. The compressive strength measured therefore may depend on the loading conditions. A detailed description of the compressive test is given in.[3] Some compressive strength results are presented in Table 1. The ratio between compressive and tensile strength ranges between 2.5 and 18 depending on the material and the porosity. Dense materials have a higher ratio than porous materials.

7.2 Global Multiaxial Fracture Criterion

Whereas the strength of materials is measured under uniaxial tension or compression loading, multiaxial stresses very often occur in components. This is the case for uniaxial loading of notched components, for components under internal pressure, or components under inhomogeneous temperature distribution. The degree of multiaxiality is described by the ratios of

$$\alpha = \frac{\sigma_2}{\sigma_1} \qquad \beta = \frac{\sigma_3}{\sigma_1} \tag{21}$$

where σ_1, σ_2, and σ_3 are the principal stresses. Two different approaches exist to assess multiaxial stresses: A global multiaxiality and a local multiaxiality criterion.

A global multiaxiality criterion is a relation between the principal stresses and one or two material properties. The most reliable criterion is Mohr's hypothesis, which is presented here for a plane stress situation. It is based on the tensile strength σ_{ct} and compressive strength σ_{cc}. The criterion is shown in Fig. 7 and reads

$$\begin{aligned} \sigma_1 &= \sigma_{ct} && \text{for } \sigma_1 > 0,\ \sigma_2 > 0 \\ \sigma_2 &= -\sigma_{cc} && \text{for } \sigma_1 < 0,\ \sigma_2 < 0 \\ \sigma_1 &= \sigma_{ct}(1 + \sigma_2/\sigma_{cc}) && \text{for } \sigma_1 > 0,\ \sigma_2 < 0 \end{aligned} \tag{22}$$

7.3 Local Multiaxiality Criterion

The local multiaxiality criteria start with a flaw type that may be a pore or a crack. The local fracture criterion is a critical tensile stress for a pore or a critical stress intensity factor for a crack. Details can be found in Ref. 2. In the pore models only the tensile strength appears as a material property. For a spherical pore the failure relation is

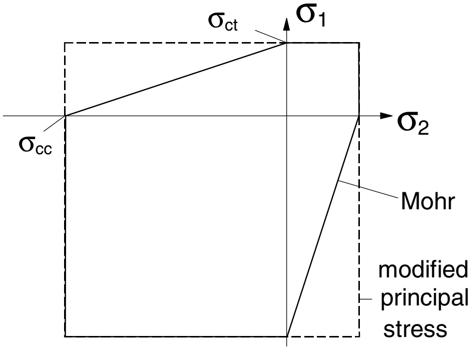

Fig. 7 Mohr's hypothesis for biaxial loading.

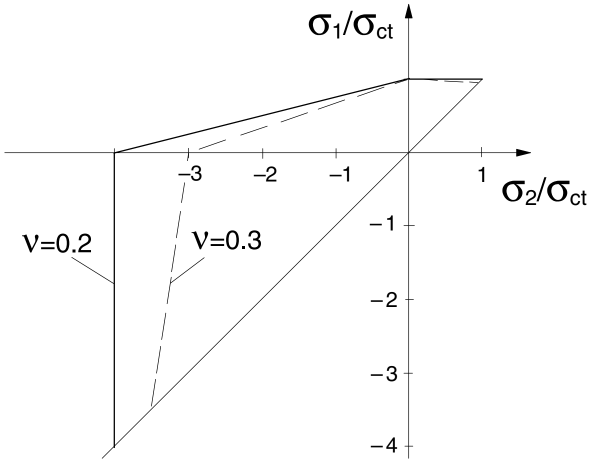

Fig. 8 Failure diagram for a spherical pore.

$$\sigma_1 + \frac{15\nu - 3}{27 - 15\nu}\,\sigma_2 - \frac{3 + 15\nu}{27 - 15\nu}\,\sigma_3 = \sigma_{ct} \tag{23}$$

From this relation the compressive strength follows as

$$\sigma_{cc} = \frac{27 - 15\nu}{3 + 15\nu}\,\sigma_{ct} = \begin{cases} 4\sigma_{ct}, & \text{for } \nu = 0.2 \\ 3\sigma_{ct}, & \text{for } \nu = 0.3 \end{cases} \tag{24}$$

Under triaxial compression, no failure is expected because the stresses at the pore are negative. In Fig. 8 the failure diagram for plane stress is shown. Results for an ellipsoidal pore are represented in Fig. 9. From these results it can be

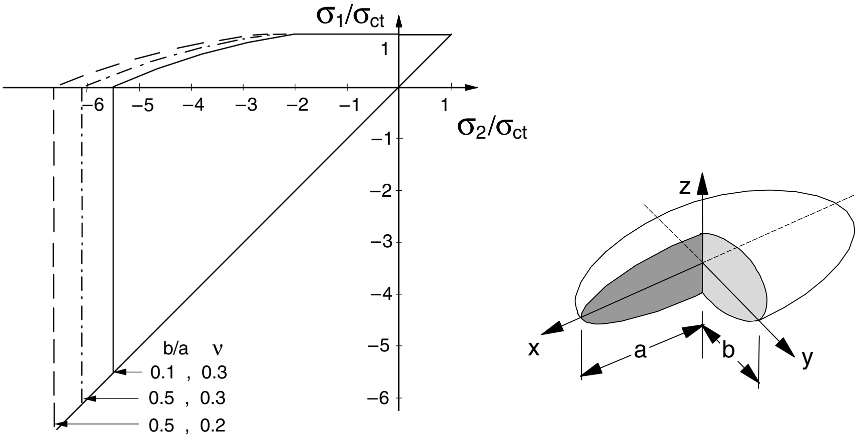

Fig. 9 Failure diagram for an elliptical pore. (From Ref. 2.)

concluded that the ratio of compressive to tensile strength may vary between about 3 and 6, depending on the shape of the pore and the Poisson ratio. It also shows that Mohr's hypothesis is a good approximation to the more detailed pore models.

For a given pore geometry (spherical or ellipsoidal with a given ration of b/a), the strength is independent of the size of the pore. Consequently, it is not possible to derive a statistical multiaxial failure relation. Such a relation can be obtained for a crack model. In this model the flaws are described as cracks with a given shape, e.g., as circular cracks or semicircular surface cracks. Then, a statistical multiaxial failure criterion can be developed in the following steps. The cracks are assumed to be randomly orientated and have a size distribution that leads to the Weibull distribution of the strength under uniaxial loading. Depending on the orientation of the flaw with respect to the principal stress axis and the ratios of the principal stresses α and β, the stresses at the crack tip are described by the mixed mode stress intensity factors K_{I}, K_{II}, and K_{III}: K_{I} is caused by the normal stress on the crack area σ_n; K_{II} and K_{III} are caused by the shear stress τ acting on the crack plane. The local failure criterion is a relation between these three stress intensity factors and the fracture toughness K_{IC}. Different relations are discussed in literature, some of them also include the fracture toughness K_{IIC} for mode II loading. An example is the criterion of constant energy release rate:

$$K_{\mathrm{I}}^2 + K_{\mathrm{II}}^2 + \frac{1}{1-\nu} K_{\mathrm{III}}^2 = K_{\mathrm{IC}}^2 \tag{25}$$

For any local stress state a critical orientation of the crack exists. If the normal stress is compressive ($\sigma_n < 0$), then $K = 0$ and K_{II} and K_{III} have to be calculated with an effective shear stress:

$$\tau_{\mathrm{eff}} = \begin{cases} |\tau| + \mu\sigma_n & \text{for } |\mu\sigma_n| < \tau \\ 0 & \text{for } |\mu\sigma_n| > \tau \end{cases} \tag{26}$$

with the friction coefficient μ. As an example, the resulting failure criterion is shown for circular cracks and a specific mixed mode failure criterion in Fig. 10.

If the scatter of the strength is included in the calculation procedure, the resulting failure criterion also depends on the Weibull parameter m. For details see Ref. 2.

For an inhomogeneous stress distribution the failure probability of a component is given by the integral of

$$F = 1 - \exp\left[-\frac{1}{2\pi}\left(\frac{\sigma_c^*}{\sigma_{\mathrm{I0}}}\right)^m \iint h^m \sin\Phi \, d\Phi \, d\Psi \int g^m \, dV\right] \tag{27}$$

The angles Φ and Ψ characterize the orientation of the crack normal to the principal stress axis. The quantity h depends on the stress ratios α and β and on the mixed-mode fracture criterion; σ_{I0} is a material-dependent critical stress.

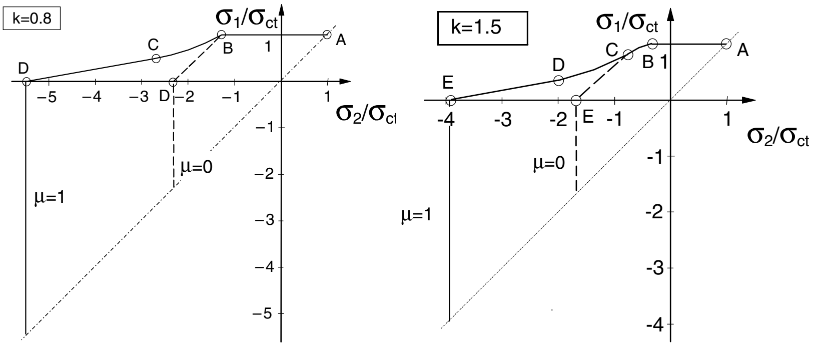

Fig. 10 Failure diagram for circular cracks (*k* characterizes the mixed mode criterion). (From Ref. 2.)

The flowchart for the computation of the failure probability is presented in Fig. 11. It shows the necessary input information:

- The stress state in the component (usually from finite-element calculation)
- A crack model
- A local failure criterion
- The material properties *m* and σ_{I0}

Postprocessor programs are available for the calculation of failure properties.

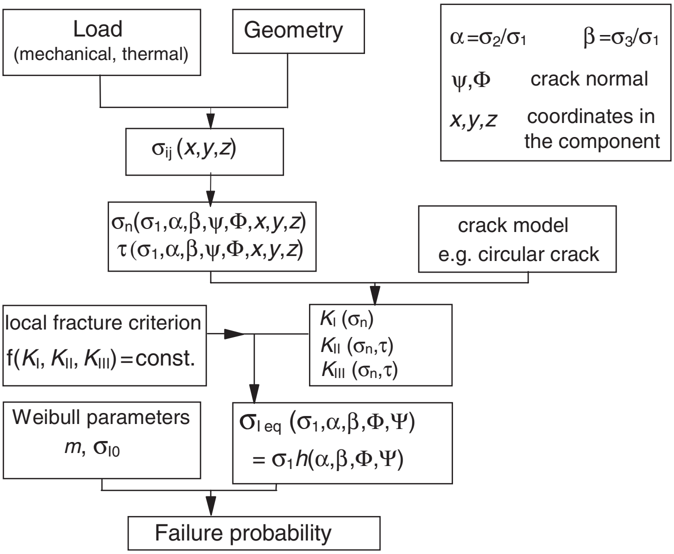

Fig. 11 Flowchart for computation of failure probability for multiaxially loaded components. (From Ref. 2.)

Summary

- The failure probability of components under multiaxial loading can be calculated applying multiaxial Weibull theory, where the flaws are described as randomly orientated cracks.
- As a global multiaxial fracture criterion Mohr's hypothesis can be applied.

8 MATERIALS SELECTION FOR THERMAL SHOCK CONDITIONS

Ceramic materials may fail during rapid cooling or rapid heating. Due to the time-dependent transient temperature distribution in a component, the thermal expansion depends on the location and has to be compensated by mechanical deformation. Whereas in metals these strains lead to small local plastic deformation, they may cause high local stresses in ceramics that may exceed the local strength. The failure probability of a component under thermal shock conditions can be calculated applying the multiaxial Weibull statistics described in Section 7.

The resistance against thermal shock varies for different materials. It depends on the following material properties: Thermal expansion coefficient α, elastic properties Young's modulus E, Poisson's ratio ν, thermal conductivity λ, density ρ, head capacity C_p, and strength σ_c. Under different thermal conditions different thermal shock parameters have been defined, which characterize the thermal shock sensitivity.

For perfect heat transfer from the medium to the component the thermal stress at the surface after a thermal shock by ΔT is

$$\sigma_{\max} = \frac{E\alpha\,\Delta T}{1 - \nu} \tag{28}$$

The critical temperature for fracture under thermal shock therefore is

$$R = \frac{\sigma_c(1 - \nu)}{E\alpha} \tag{29}$$

For cooling with a constant heat transfer coefficient h the maximum stress at the surface increases with time and decreases after passing a maximum. The maximum stress is given by

$$\sigma_{\max} = \frac{E\alpha\,\Delta T}{1 - \nu} f(B) \tag{30}$$

where f is a function of the Biot modulus

$$B = \frac{hd}{\lambda} \tag{31}$$

where λ is the thermal conductivity, and d a characteristic size parameter of the component. For small values of B the maximum stress is proportional to B. Hence the critical temperature for failure is proportional to

$$R' = \frac{\sigma_c \lambda (1 - \nu)}{E\alpha} \tag{32}$$

For a constant heating rate at the surface the maximum stress is

$$\sigma_{\max} = -C \frac{\alpha E \rho C_p}{\lambda(1 - \nu)} \frac{dT}{dt} \tag{33}$$

where ρ is the density, C_p the heat capacity, and C a constant depending on the geometry. The critical heating rate therefore is proportional to

$$R'' = \frac{\sigma_c \lambda (1 - \nu)}{E\alpha\rho C_p} \tag{34}$$

Whereas under mechanical loading a crack extends unstably after initiation, a crack stops after some extension under thermal shock conditions. The remaining strength, however, decreases considerably. If the strength of the component is not of importance, a limited amount of crack extension may be tolerable. Then, the condition for materials selection may be a small extension of an existing crack. It can be shown that the amount of crack extension during thermal shock increases with increasing crack size. Consequently a material with a large initial crack may be of advantage. The initial crack size follows from

$$\alpha_i = \left(\frac{K_{\mathrm{IC}}}{\sigma_c Y} \right)^2 \tag{35}$$

leading to another thermal shock parameter

$$R'''' = \frac{K_{\mathrm{IC}}^2}{\sigma_c^2} \tag{36}$$

(a R''' is not considered here).

In Table 5 typical values of the thermal shock parameters are given.

Summary

- The sensitivity to failure by thermal shock increases with increasing Young's modulus, increasing thermal expansion coefficient, decreasing strength, and decreasing thermal conductivity.
- For a constant heating rate at the surface the sensitivity decreases further with increasing density and increasing heat capacity.

Table 5 Thermal Shock Parameters for Different Materials

					Si_3N_4			
	Al_2O_3	MgO	ZrO_2	SiC	HPSN	RBSN	BeO	Al_2TiO_5
α (10^{-6} K^{-1})	8	12	11	4	3.2	2.5	8	1.8
E (GPa)	400	270	200	350	300	180	360	30
ν	0.22	0.17	0.25	0.2	0.28	0.23	0.25	0.2
λ (W m^{-1} K^{-1})	30	30	2.5	100	35	10	300	2.5
ρ (g cm^{-3})	3.9	3.5	6.0	1.0	0.7	0.7	1.3	0.7
C (J g^{-1} K^{-1})	1.0	1.0	0.5	1.0	0.7	0.7	1.3	0.7
σ_c(MPa)	300	180	950	360	660	200	180	65
K_{IC} (MPa $m^{1/2}$)	4.5	3.0	10	4	7	2	4.8	—
R (K)	73	46	324	206	495	342	47	962
R' (kW m^{-1})	2.19	1.38	0.81	20.6	17.3	3.42	14.1	2.41
R'' (W cm^2 g^{-1} K)	5.6	3.9	2.7	66	75	20	36	9.6
R'''' (mm)	0.23	0.28	0.11	0.12	0.11	0.10	0.71	

9 FAILURE AT HIGH TEMPERATURES

Failure at high temperatures is caused by creep elongation and creep rupture. Another failure mode is oxidation. Different mechanisms may contribute to creep and creep damage. In many cases, creep is caused by grain boundary effects, such as viscous flow of an amorphous grain boundary phase, diffusion of vacancies along grain boundaries, formation and extension of cavities at grain boundaries or dissolution, and reprecipitation of material through the glassy phase. Creep within the grain is caused by the motion of dislocations or vacancies.

Damage mechanisms leading to the rupture of a component can be the formation of grain boundary cavities, coalescence to cracks, and growth of the cracks until final failure.

9.1 Creep Strain

After application of a load to a specimen, the same variation of strain with time as in metals is observed. After an instantaneous strain ε_0, the creep curve can be subdivided into three stages. In stage I—the primary creep—the strain rate decreases and reaches in stage II—secondary creep—a constant value. Afterwards, in stage III—tertiary creep—the strain rate increases.

Primary creep can be described by one of the following relations:

$$\varepsilon_p = At^m \quad \text{with } m < 1 \tag{36a}$$

$$\varepsilon_p = A[1 - \exp(-mt)] \tag{36b}$$

The effect of the stress on the creep rate very is often described by Norton's law:

$$\dot{\varepsilon} = B\sigma^n \tag{37}$$

Other proposed relations are

$$\dot{\varepsilon} = B[\exp(\alpha\sigma) - 1] \tag{38}$$

$$\dot{\varepsilon} = B \sinh(\alpha\sigma) \tag{39}$$

These relations can be applied for the primary and secondary creep range with possibly different parameters α and n.

Creep under compression differs from creep in tension especially for secondary creep, where the creep rate in compression is lower than under tension. An example is shown in Fig. 12. In this case the exponent n in Eq. 37 is similar in tension and compression, the constant B, however, is different. Figure 12 also shows that Eq. 37 cannot be applied for the whole range of the stress: The exponent n for small stresses is lower than at higher stresses.

The effect of temperature usually can be described by

$$\dot{\varepsilon} = C \exp\left(-\frac{Q}{RT}\right) \tag{40}$$

where R is the gas constant and Q the activation energy for the leading creep process. An example of Eq. 40 is shown in Fig. 13.

The problem of these relations is that the lowest creep rates measured are usually higher than the allowable creep rate in a component. It is obvious from Fig. 12 that the lowest creep rates measured in these investigations are in the order of 10^{-9}/s. For this creep rate the creep strain is 3% in one year. Therefore an extrapolation to lower creep rates often is necessary.

9.2 Creep Rupture

The mechanism of creep rupture differs from the mechanism of subcritical crack extension at low temperatures. This can be seen in Fig. 14, where the stress is plotted versus the lifetime for an alumina at 1100°C in a log-log plot. Two ranges can be distinguished. At high stresses, the failure is caused by the extension of preexisting flaws. The relation between stress and failure strain can be described

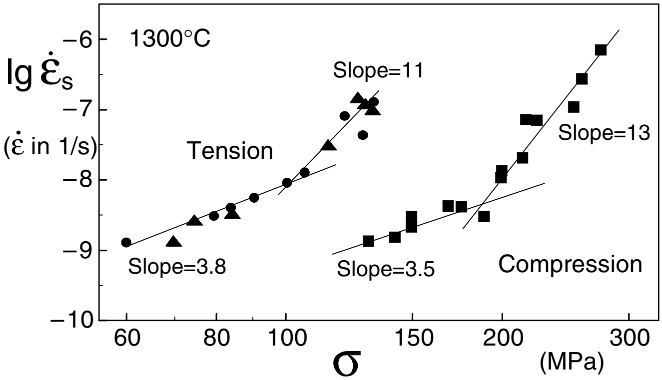

Fig. 12 Stress dependence of the secondary creep rate for reaction-bonded siliconized silicon carbide tested under tension and compression (from Ref. 4).

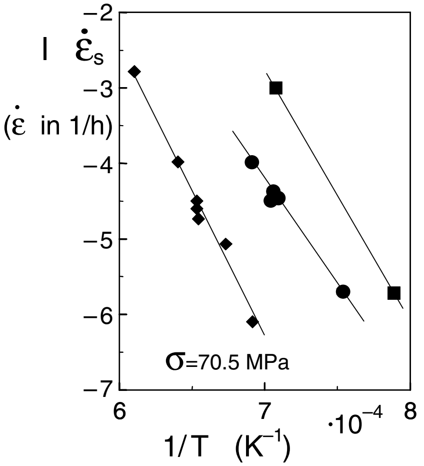

Fig. 13 Secondary creep rate as a function of temperature for three hot-pressed silicon nitrides. (From Ref 5.)

by Eq. 3a with $n = 12$ [the slightly curved line in Fig. 14 is obtained by applying Eq. 3a for the bending tests taking into account small stress redistribution due to creep]. At lower stresses, the observed lifetime is much lower than predicted from Eq. 3.

Summary

- Creep at high temperatures is described by relations between creep rate, stress, and temperature.
- The time to failure in the creep range is less stress dependent than at lower temperatures.

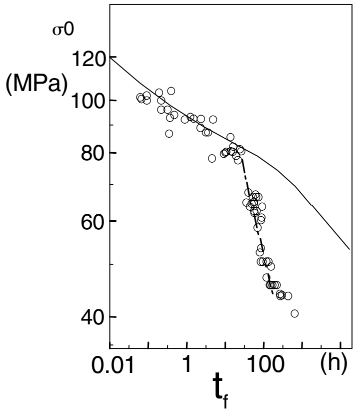

Fig. 14 Lifetime as a function of the (elastically calculated) outer fiber bending stress for static bending tests on Al_2O_3 with 4% glass content. Solid line predictions from subcritical crack growth.

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CHAPTER 28

MECHANICAL RELIABILITY AND LIFE PREDICTION FOR BRITTLE MATERIALS

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1 SCOPE

This chapter is intended to provide the reader with a general understanding of why brittle materials fail in a time-dependent manner in service and how to estimate the lifetimes that can be expected for such materials. In addition, we describe procedures to evaluate the confidence with which these lifetime predictions can be applied. Throughout this chapter, we assume that the material under investigation can be treated as isotropic and homogeneous, and that microstructural influences on properties can be ignored. Therefore, more complicated issues, such as crystallographic texture, grain-boundary phases, and R-curve behavior, lie outside the scope of this discussion and will not be addressed herein.

2 INTRODUCTION

Techniques to determine reliability of components fabricated from brittle materials (e.g., ceramics and glasses) have been extensively developed over the last 30 years.[1–7] Reliability is generally defined as the *probability* that a component, or system, will perform its intended function for a specified *period of time*.[8] Accordingly, the two overarching principles influencing reliability are the statistical nature of component strength and its time-dependent, environmentally en-

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hanced degradation under stress. The statistical aspect of strength derives from the distribution of the most severe defects in the components (i.e., the strength-determining flaws).[9–15] The time-dependent aspect of strength results from the growth of defects under stress and environment, resulting in time-dependent component failure.[16–19] These concepts have lead to a lifetime prediction formalism that incorporates strength and crack growth as a function of stress. Predicted reliability, or lifetime, is only meaningful, however, when coupled with a confidence estimate. Therefore, the final step in the lifetime prediction process must be a statistical analysis of the experimental results.[2,20–22]

While phenomenological, the reliability methodology has been useful in predicting lifetimes for myriad applications, including all-glass aircraft windows,[22] spacecraft[23,24] windows, flat panel displays,[25] optical glass fibers,[26] porcelain insulators,[27] vitreous grinding wheels,[28] and electronic substrates.[29] Moreover, although developed for isotropic and homogeneous materials, the methodology appears to be generally valid for most fine-grain ceramic materials,[7,30–32] and perhaps for single-crystal materials.

The lifetime prediction approach, described below, represents the currently accepted procedures of reliability assessment for homogeneous, brittle materials. There are, of course, a number of assumptions built into any technical procedure and, frequently, these assumptions cannot be tested. The assumptions inherent in steps associated with lifetime prediction are clearly stated in this chapter, and implications that arise if the assumptions are violated are discussed at the end of each section.

The structure of this chapter has been chosen to allow the user to access easily regions of particular interest. It has been arranged to provide a general overview of the processes involved in lifetime prediction of brittle materials followed by a series of appendixes that describe the technical details and requirements for each of the different stipulated experimental procedures. The overview describes why the various measurement procedures are necessary and how they fit into an overall lifetime prediction model. For details associated with the derivation of the models, the user is directed to the references at the end of the chapter.

3 OVERVIEW

General Considerations

The most basic assumption made in this chapter is that the material whose lifetime is of interest is truly brittle; that means there are no energy dissipation mechanisms (e.g., plastic deformation, internal friction, phase transformations, creep) other than catastrophic bond rupture occurring during mechanical failure. It has been well documented that brittle materials fail from flaws that locally amplify the magnitude of stresses to which the material is subjected.[9,10,14,15,33] These flaws, e.g., scratches, pores, pits, inclusions, or cracks, result from processing, handling, and use conditions. For a given applied or residual stress, the initial flaw distribution determines whether the material will survive application of the stress or will immediately fail. Similarly, the evolution of the flaw population with time determines how long the surviving material will remain intact.

Other key assumptions in reliability predictions for brittle materials are that the experiments used to determine inert strength distributions do not alter the initial flaw population and that the flaw population in the test pieces mimics that in the final component. In addition, we assume that the flaws change only through the crack growth processes described in the model. It is essential, therefore, that the test specimens that are used to determine the initial flaw distribution be fabricated by the same processing conditions and prepared with the identical surface preparation as applied to the final components.[4,34] It is also essential that care be taken to avoid damaging test specimens during fabrication, storage, and handling. The care taken often includes, but is not limited to, chamfering test specimens to reduce failures resulting from edge defects, individually wrapping test specimens to prevent damage during storage and transportation, and avoiding contact with the tensile surface of the specimens.

Strength

Because the flaw population under stress defines the initial strength of a brittle material, it is necessary to characterize this distribution or, equivalently, the distribution of initial strengths. Almost always, the initial strength distribution is easier to characterize. This can be done in three ways: (1) a statistical characterization of the initial strength distribution, (2) an overload proof test to establish a minimum strength, or (3) direct nondestructive flaw detection from which strength can be calculated. Each of these characterization techniques, described below, has specific advantages. However, each of the techniques also contains stringent requirements and limitations. It is essential to bear in mind that if these requirements are not met or the limitations are exceeded, the techniques will lead to erroneous lifetime predictions.

Statistical Strength Distribution

The first strength characterization technique to be discussed is the statistical strength characterization approach. This approach is particularly useful to evaluate the suitability of different materials, processing procedures, and surface treatments for a given application. However, because the approach is statistical, it cannot guarantee that components will have strengths greater than some minimum value; rather, it provides a failure (or survival) *probability* for a given load level.

Because it is the most critical flaw in a component or test specimen that leads to failure, it is not the distribution of flaws itself that is important but rather the distribution of the extremes of the flaw distribution. Fortunately, distributions of extremes, or extreme-value statistics, have been thoroughly studied.[11–13,35] Strength distributions are typically described by the Weibull distribution, one of three extreme-value distributions. Procedures for estimating the parameters of the Weibull distribution are well documented.[36–38] The generally accepted procedure is to use maximum likelihood estimators. Procedures for the two-parameter Weibull distribution are described in the ASTM standard practice guide, ASTM C 1239-95.[36] Occasionally, the strength data exhibits threshold-strength behavior, and must be fitted to a three-parameter Weibull distribution.[22] In such cases a number of difficulties are associated with the numerical techniques used to estimate the Weibull parameters.[37,38] Typically a Newton-Raphson iteration procedure is used (see Appendix F of Ref. 37), but a sequential search technique with subsequent bisectional bracketing also works well.[22]

The strength distribution obtained is unequivocal provided that it is determined on actual components (for each batch of material) under conditions that simulate the service conditions. However, this requirement often poses an intolerable economic constraint, because so many tests must be performed to achieve an acceptable confidence level. Consequently, tests are often conducted on small coupons whose processing history and surface treatment mimic those of the final component.

Some care must be exercised in collecting the strength data. Measurements should be conducted at a high loading rate and in an inert environment, e.g., a dry gaseous nitrogen environment, to avoid environmental effects discussed below. In addition, the stress state, σ, used for the Weibull test must represent as closely as possible the stress conditions the component will see in service. For example, conducting the Weibull tests in uniaxial tension will not necessarily provide meaningful data if the actual component will experience biaxial loading.

After the Weibull strength distribution has been obtained and analyzed, a particular probability of failure, F, can then be associated with a corresponding initial strength, S. The relationship between F and S, i.e., the spread of initial strengths, is controlled by the distribution of initial flaw sizes in the material.

Finally, if the tests have been conducted on test coupons rather than final components, it is essential to relate the area under load in the coupon to the area under load in the component (see Appendix 1) because the probability of encompassing a more severe flaw is proportional to the area under load. Therefore, the test specimen area under load must be normalized to the component area under load. It should be borne in mind that, if failure results from subsurface defects, the important parameter to normalize is the volume under load rather than the surface area.

Minimum Strength Overload Proof Test

An overload proof test is a procedure for establishing the upper limit on the most critical flaw in a component or, equivalently, *the lower limit on the initial strength distribution*.[4,7,39–43] The technique is expensive since real components are tested. However, a properly conducted proof test completely eliminates the problem of failure through statistical outliers that is unavoidable when the statistical strength distribution technique is used. Therefore, if the statistical strength distribution analysis indicates that there is an unacceptably high probability of failure at the required load, the overload proof test procedure can be used to eliminate the weak components and truncate the failure distribution. It should be noted that, because the overload proof test is applied to finished components, it is generally not a technique that is appropriate for material or design selection. In addition, for a final component with a complicated geometry, it can be difficult to apply controlled stresses that are equivalent in orientation to those experienced in service.

In concept, the overload proof test is straightforward. One simply applies an overload stress to each component, typically two to three times the service stress. The ratio of the overload stress to the service stress is the *proof-test ratio*. Components with critical flaws larger than a predetermined size, or with strengths less than a predetermined minimum (namely, the proof-test stress), will break. Accordingly, such components are automatically eliminated from the distribution.

However, the requirements for a properly conducted proof test must be stringently met. Specifically, the proof test must be devised such that the stress at each location of a component exceeds the service stress in that same location by an amount at least as large as the recommended proof-test ratio. The proof test must be performed under controlled conditions of environment and proof-test load cycle; the most critical aspect being to have conditions as inert as possible and to unload from the proof stress as rapidly as possible. If these conditions are satisfied, then proof testing is undoubtedly the best lifeprediction procedure, because no aspects of the analysis are unresolved. If the conditions are not met, then crack growth (discussed below) could occur during unloading, obviating the desired truncation of the initial flaw population and leaving the components weaker than predicted by the proof test-analysis.

Nondestructive Flaw Detection

In principle, a good nondestructive evaluation (NDE) test for flaw detection offers several benefits: 100% inspection, on-line inspection, in-field health monitoring, and quantitative critical flaw detection. In practice, the approach is almost never used for brittle materials. The difficulties associated with using NDE involve both flaw detection and flaw quantification. Since the toughness of most ceramics and glasses is on the order of 1–5 $\text{MPa}\cdot\sqrt{\text{m}}$, critical flaw sizes are less than approximately 50 μm for even low-strength materials. This is currently beyond the detection limit of most available NDE procedures. However, measurement techniques are rapidly improving and, within the foreseeable future, instruments may be commonly available that will detect defects of micrometer and sub-micrometer size. A more formidable task will then be relating the NDE measurements to quantifiable reliability predictions.

As discussed above, brittle materials fail from the most severe, not the average, flaws. The phrase "most severe flaw" means the flaw that generates the largest stress intensity in the expected stress field. Consequently, the flaw size, shape, and orientation relative to the stress field must be quantified before results from an NDE measurement can be used to predict strength.

Unlike metals, in which flaws can grow to many times their original size without component rupture while NDE techniques monitor the flaw growth or the concomitant material property degradation, brittle materials fail with very limited flaw extension and almost no property degradation. Consequently, unless a specific NDE test has been clearly shown to predict failure strength accurately for the same brittle material with the same surface finish and under the same loading conditions of interest, it is better to use the statistical strength distribution or the overload proof-test techniques described previously to determine strength information.

Environmentally Enhanced Fracture

The second property influencing the reliability of brittle materials is the growth of flaws in the presence of stress. If the flaws remained constant with time,* there would be no need to proceed beyond the initial strength analysis. Based

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*Throughout this discussion, we consider only the *growth* of preexisting flaws over time and ignore the possibility of generating a new flaw population during use.[4,34]

upon the Weibull distribution, the probability of failure would be known for any stress condition and would be constant. Alternatively, based on an overload proof test or NDE, a minimum strength is defined such that failure would not occur for stresses below these values. However, it is well known that flaws grow under stress in most brittle materials.[1,16–19,44,45] As the flaws grow, the material weakens, leading to a time-dependent mechanical failure. This process is known by several names; three of the most common are "environmentally enhanced fracture," "delayed failure," and "static fatigue."

Environmentally enhanced fracture is a process that is still not completely understood. However, it is known that flaws in materials under stress can react with certain environments, resulting in bond rupture and flaw growth at stresses well below the nominal bond strength of the material. Water is particularly effective at enhancing fracture in many brittle materials. In addition, water is almost ubiquitous in either liquid or vapor phase. Therefore, it is essential that the effects of water-enhanced fracture be incorporated into any lifetime prediction model.*

Because the process of environmentally enhanced fracture is only partially understood, expressions, relating crack growth velocity, V, to stress intensity factor, K_I, are typically phenomenological rather than truly theoretical. Although several crack-growth-rate expressions have been proposed, the one used in almost all lifetime predictions for brittle materials is given by:[2–4,6,7,39]

$$V = V_C \left(\frac{K_I}{K_{IC}}\right)^N \tag{1}$$

where V_C is a constant and N is called the environmentally enhanced crack growth parameter, sometimes denoted the inverse crack growth susceptibility. K_{IC} is the critical stress intensity factor at which fracture ensues catastrophically but, for the present context, can be considered as a scaling parameter for the stress intensity factor, K_I. N denotes the extent to which the environment interacts with the stressed crack to cause enhanced crack growth. For a given V_C and K_{IC}, the larger N is, the less effective the environment is at enhancing crack growth.

The fact that Eq. 1 is phenomenological rather than rigorously based on theory should not be a concern as long as the equation describes the observed behavior, *particularly in the low stress region.* Guaranteeing this condition is difficult, however, since the crack growth rates for a component in use are usually several orders of magnitude smaller than the lowest crack growth rates that can be determined experimentally. As long as any deviation from Eq. 1 is of the form $V_{\text{real}}(K_I) < V_{\text{Eq. 1}}(K_I)$, that is, the real velocity is less than the velocity predicted from Eq. 1, lifetime predictions based upon Eq. 1 are conservative. However, if $V_{\text{real}}(K_I) > V_{\text{Eq. 1}}(K_I)$, lifetime predictions based on Eq. 1 are no longer conservative.

At this point, it should be noted that there are a number of other possible crack growth expressions that are statistically indistinguishable from Eq. 1 over

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*Few environments are more effective than water in enhancing crack growth for most brittle materials. An exception is very basic solutions for silica-based glasses.[45] For such cases, the following discussion still applies, but the tests must be conducted in the new environment.

the measurable crack velocity range.[46] Some of these expressions are more conservative, i.e., predict higher crack growth rates, at low K_I than the power law. A particularly important example is the expression $V = B \exp[bK_I]$, which is derived from chemical rate theory, and has been used by several authors to parameterize environmentally enhanced fracture.[3,24,46–48] Although having a stronger scientific basis than the power law (Eq. 1), the exponential expression for crack growth rates has never developed the following obtained by the power law expression. The dominance of the power law expression derives from the fact that it allows closed form, analytical lifetime solutions to be obtained even when the critical flaws are surrounded by a residual stress field, as will be discussed in the section on constant loading rate experiments. With the advent of powerful desktop computing, it is now possible to use numerical techniques, rather than analytical derivations, to predict lifetimes. The approach outlined above is still required, i.e., determination of the initial strength distribution through Weibull statistics[36–38] or proof test[4,39] measurements, evaluation of crack growth parameters through dynamic fatigue experiments,[4,49] and estimation of confidence limits through statistical analysis, e.g., bootstrap methods[21,22,50–52] as discussed below. Consequently, it is now possible to make lifetime predictions using crack growth relationships like the exponential expression. However, it is much easier to demonstrate how the different measurements are combined to obtain an expression for the lifetime if analytical expressions are used. Therefore, for the purposes of this chapter, we will focus exclusively on the power law expression for crack growth.

Constant Loading Rate Experiments

Conducting fracture mechanics crack growth experiments to evaluate the parameters governing environmentally enhanced crack growth is a process that requires both a large amount of time and a thorough understanding of issues associated with environmentally enhanced fracture. Consequently, a faster evaluation method that is less prone to experimental subtleties is desirable. Equation 2 defines the constant loading rate or dynamic fatigue measurement relationship (Appendix 2). In Eq. 2, λ is a fitting parameter related to V_C and K_{IC} and $N' = (3N + 2)/4$, where V_C, K_{IC}, and N are parameters in Eq. 1.

$$\sigma_f^{N'-1} = \lambda \frac{d\sigma}{dt} \tag{2}$$

Although there are various possible geometries and several critical experimental requirements for dynamic fatigue measurements (see Appendix 2), the measurements are conceptually straightforward. A series of specimens is subjected to a load that increases linearly with time. The log of the failure strength, σ_f, plotted as a function of stressing rate, σ, gives the parameters N' and λ. To obtain conservative values of N' and λ, dynamic fatigue measurements need to be carried out in the harshest environmental conditions under which the final component will be used. For example, if water vapor enhances crack growth, liquid water could provide the limiting case of water-enhanced environmental fracture. However, it is not necessary that the initial flaw distribution be maintained. A change in flaw distribution will change σ_f, but not the slope, $\lambda/(N - 1)$. Therefore, dynamic fatigue experiments are usually conducted on indented specimens;

the indentations provide a uniform and repeatable critical flaw.[53–57] Typically, the resultant data have much less scatter than those obtained from the initial flaw distribution, thereby resulting in a more precise determination of the slope.

Inert Strength for Indented Specimens

The inert strength for indented specimens, S_v, is determined using the same procedure as that used to obtain the Weibull distribution: high loading rate and an inert atmosphere. However, instead of using the natural flaw population as the strength limiting defects, S_v uses the same level of indentations that were used in the dynamic fatigue experiments. These data provide an upper limit on the dynamic fatigue strength data.

Lifetime Prediction

Once the Weibull distribution, the dynamic fatigue data, and the inert strength for the indented specimens have been obtained, the lifetime, t_f, for any initial strength, S, and any applied stress, σ, can be calculated using Equation 3[22]:

$$t_f = \frac{\lambda}{N' + 1} \left(\frac{S}{S_v} \right)^{N'-2} \sigma^{-N'} \tag{3}$$

Although Eq. 3 predicts a time to failure for a component under load, there is still no way to estimate how statistically significant the value is. To obtain the most information from the lifetime prediction model, it is necessary to estimate the confidence limits surrounding t_f.

Confidence Limits

As with any experimentally determined quantities, there are uncertainties associated with each of the parameters in Eq. 3. Some of the parameters may not be amenable to standard error propagation analysis. For example, for large N', e.g., $N' \geq 60$, the uncertainties in evaluating the slope of the dynamic fatigue curve are much larger than the calculated slope itself. Under such conditions, standard error propagation techniques are invalid. An alternative is the bootstrap technique.[50–52] It is a statistical procedure that takes advantage of modern computing power and makes it relatively straightforward to determine confidence limits (Appendix 3). It applies to any data set that is *independent and identically distributed* (iid). The major assumption with the bootstrap technique is that the data used to evaluate the parameters in Eq. 3 adequately represent the statistical scatter in the experiments. If this condition is met, combining the bootstrap technique with the data used to evaluate t_f permits the user to estimate the time to failure at whatever confidence level is desired.

Life Prediction Process

The individual steps required for lifetime prediction have been laid out above. If the steps are followed and careful attention is paid to the requirements and restrictions at each step in the process, a lifetime prediction with a specific failure probability and known confidence limits for a specific applied load is obtained. However, frequently, other questions are of interest. For example, the needed lifetime may be an input parameter, and the limiting stress value may be the unknown parameter of interest. As another example, a required lifetime at a known load may be the input parameters, and the unknown may be whether a given material can be used and, if so, with what probability of failure. The formalism outlined above is adequate to address any of these questions and, in fact, can be used in an iterative manner to determine how variations in one group of parameters affect others. In this section, failure diagrams will be introduced. These diagrams, constructed from the data obtained using the techniques outlined above, graphically relate the parameters to show quantitatively how modifications in one parameter, e.g., stress or lifetime, affect the others.

Figure 1*a* is derived directly from Eq. 3. Each of the solid lines maps the applied stress, σ, onto the time to failure, t_f, for a different value of the initial strength or, equivalently, different probability of immediate failure upon the application of the σ (see Appendix 1). The lines are all parallel with slope of $-N'$. The bands about each line represent the confidence limit at which the position of the line is known and can be determined by a statistical analysis tool such as the bootstrap method. For conservative calculations, the left-hand confidence limit should be used instead of the centerline. Figure 1*a* immediately shows, in a quantitative way, how all the parameters affecting lifetime predictions are coupled if a statistical strength distribution is being used. Known, or required, values of some of the parameters can be located on the plot and the range of possible values for the remaining parameters can be taken directly from the graph.

If the overload proof test, rather than the statistical strength distribution, approach is being used, Fig. 1*b* maps the applied stress onto the lifetime. This figure is obtained from Eq. 3 by replacing the initial strength S divided by σ by the proof-test ratio, R. This gives a linear relationship between $\log(\sigma)$ and $\log(t_f)$ with a slope of -2. The different parallel lines in Fig. 1*b* are now determined by changes in R. The larger R is, the larger t_f is for a given σ, but at the cost of more components being broken. To estimate the fraction of components that will fail during the proof test, one refers again to the probability diagram, the proportion of components likely to fail in the proof test can be estimated by determining the intersection of the stress coordinates of the proof stress, $\sigma_p = R\sigma_a$, and the time coordinate for the time at the peak proof load, t_p. If this proportion is unacceptable, e.g., most of the components fail in the proof test,

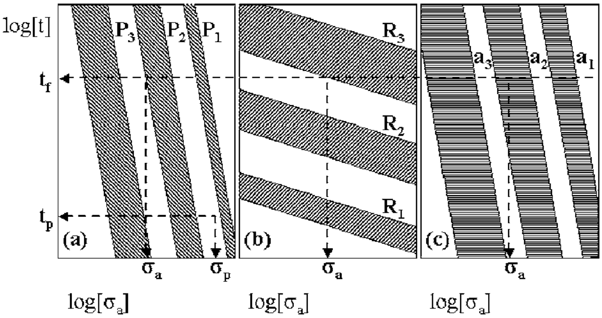

Fig. 1 Design approach.

then the material, including flaw distribution, is inadequate, and an alternate material should be explored. This process should proceed until a material with a satisfactory proof-test failure proportion is identified.

Finally, if proof testing is unsuitable, for example, as a result of the complexity of the component, NDE techniques could be explored. The approach taken at this juncture is strongly material dependent. Figure 1*c* relates the lifetime, t_f, to the applied stress, σ_a, and the flaw size, a. To use Fig. 1*c*, an approximate value for the flaw size that must be detected to ensure integrity is first obtained from the graph, without accounting for the extension of the confidence limits. Then, the ability to detect such flaw types should be evaluated using available NDE techniques. At this stage, confidence limits on flaw detection in the test material can be established for each NDE technique, and the flaw size detection requirement can be more closely defined. The suitability of the material for this application is determined by the capability of detecting *all* flaws in the critical parts of the component that exceed the specified size. As discussed previously, use of this approach requires that NDE techniques are available that provide quantitative size and shape data regarding flaws.

4 SUMMARY

In summary, the following procedure is required to predict lifetimes in isotropic, homogeneous, brittle materials:

1. Fast fracture tests in an inert environment to determine the Weibull distribution of the initial flaws/strengths and the probability of failure for any arbitrary initial strength, *or* a proof test to weed out specimens below a predetermined initial strength, *or* an NDE technique that guarantees a lower limit on strength
2. Dynamic fatigue measurements on indented specimens to determine susceptibility to environmentally enhanced fracture
3. Fast fracture tests in an inert environment on indented specimens to determine the inert strength (strength in the absence of environmental effects) of the indented material
4. Bootstrap Eq. 3, using the data in steps 1–3, to predict lifetimes and determine confidence levels for predetermined initial strength

It is essential for the user to remember that this lifetime prediction methodology assumes that the flaw/strength distribution determined from the Weibull analysis is the same distribution to be found in the final components (for the statistical strength distribution approach), that the tests do not alter the flaw distribution, and that flaws change over time only through environmentally enhanced fracture as described by Eq. 1.

APPENDIX 1: WEIBULL TESTS

Since brittle failure is expected to occur from the most severe defect in the component, the strengths are assumed to come from an extreme value distribution. A critical assumption in the following discussion is that the strength distribution in the strength test is the same as the distribution in the final components. Typically, either a two- or a three-parameter Weibull is used to evaluate the distribution. Equation A1.1 gives the form of the three-parameter Weibull expression[8,22,35]

$$(1 - F) = \exp\left[-\left(\frac{\sigma(x) - S_t}{S_0 - S_t}\right)^m\right] \tag{A1.1}$$

Two of the three parameters in Eq. A1.1 are the shape parameter, m, which determines the shape of the probability distribution function, and the scale parameter, S_0, which defines the scale of the strength axis. The shape parameter, m, defines how quickly the probability of survival drops from a value of 1.0 as the stress on the material increases. Low values of m correspond to a broad, relatively slowly varying decrease in the probability of survival as a function of increasing σ. In contrast, high m values generate a survival probability curve approaching a step shape; the survival probability remains near 1.0 until σ nears S_0 and then drops rapidly to zero. As implied by the previous sentence, S_0 is related to the value of stress at which the probability of survival is near the midpoint between 1.0 and 0. For $S_t = 0$, the probability of survival at $\sigma = S_0$ is $1/e$. In fact, in the limit of m approaching infinity and $S_t = 0$, S_0 is identically the value of stress for which the probability of survival drops from 1.0 to 0.

The third Weibull parameter, S_t, represents a "threshold" value for strength below which there is zero probability of failure. The form of the two-parameter Weibull expression is identical with Eq. A1.1 with S_t equal to zero; i.e., there is no lower strength threshold. The decision as to whether S_t is zero or finite is not always easy to make. The question may become moot if it is borne in mind that the two-parameter distribution is a special case of the three-parameter distribution. The decision whether to include a finite value for S_t could be addressed statistically by determining the significance of a threshold value and uncertainties related to its inclusion (i.e., with what confidence can a finite value be attributed to S_t?) by the bootstrap simulation (Appendix 3). However, the implications of assuming a two-parameter or a three-parameter Weibull distribution should be kept in mind. A two-parameter distribution is always more conservative, and, therefore, safer, since it implicitly assumes that there is a possibility for failure at any load. However, if a threshold does exist, use of a two-parameter distribution will have economic consequences because it will predict a finite failure probability at loads for which no failures will occur.

Based on Eq. A1.1, a set of test specimens can be broken rapidly in bending (Appendix 2) in an inert environment and the strength of each specimen plotted against the survivability ranking, $(1 - F)$:

$$\ln\left[\ln\left(\frac{1}{1 - F}\right)\right] = m \ln[\sigma(x) - S_t] - m \ln(S_0 - S_t) \tag{A1.2}$$

This results in a line of slope m and intercept of $-m \ln(S_0 - S_t)$. Therefore, the survival probability of a given *test specimen* could be predicted for a given load.

However, usually, the area under load on a component in use is not identical to the area sampled in the bend test. Because the flaws are assumed to be distributed randomly in both the final components and the test specimens, if the area under load in the final component is larger than the area sampled by the Weibull test, the component is likely to sample larger flaws than those sampled during the tests. Therefore, the probability of failure at a given load will be greater for the component than for the test specimens. The converse is true if the area sampled in the final component is smaller than that sampled by the test specimens. Thus, a weighting factor is incorporated into the analysis to account for differences in sampling area between the test specimens and the final components.

This is handled in the following way. If an elemental area, dA, of a component surface at position x experiences a tensile stress $\sigma(x)$ and a survival probability, $(1 - F)$, then the survival probability of the component, $(1 - F_c)$, is the joint survival probability of all the individual surface elements. In the limit of infinitesimal elemental areas, this survival probability is

$$[1 - F_c(S)] = \exp\left[-k(m, S_t)\left(\frac{S - S_t}{S_0 - S_t}\right)^m\right] \tag{A1.3}$$

where F_c is the failure probability of the component, S is the maximum tensile stress in the component (i.e., the strength), and $k(m, S_t)$ is an area-scaling factor given by

$$k(m, S_t) = \frac{1}{A_0}\iint\left(\frac{\sigma(x) - S_t}{S - S_t}\right)^m dA \tag{A1.4}$$

If A_0 is chosen to make the k factor for the laboratory specimen unity, $k(m, S_t)$ is the relative area-scaling ratio between the components and the laboratory specimens.

Best Practices

- Unless there is clear evidence that a threshold exists in the Weibull distribution, it is better (i.e., conservative) to assume a two-parameter Weibull distribution.
- A good rule of thumb is that 30 or more specimens should be used to generate a Weibull plot.
- It is frequently valuable to identify the failure source of specimens under an optical microscope.[58,59] This procedure distinguishes failures from machining damage, inclusions, handling accidents, etc. A description of fractography is outside the range of this document, but an excellent discussion can be found at the web site: http://www.ceramics.nist.gov/webbook/fracture/fracture.htm.
- Measurements should be conducted in as inert an environment as possible. Dry flowing N_2 gas in a glove bag is usually a good choice.
- Measurements should be made at the highest loading rate for which the testing machine provides reliable data.

Dangers

- Modifying the flaw distribution on the specimen tensile surfaces through improper handling procedures
- Testing in such a manner (speed or environment) that allows slow crack growth to occur during the test

APPENDIX 2: STRENGTH AND DYNAMIC FATIGUE TESTS

Strength tests are tests in which the strength of specimens (final components or specially fabricated test specimens) is measured at a constant loading rate. For inert strength measurements, the loading rate is high and the specimens are tested in an inert environment to minimize slow crack growth. In contrast, dynamic fatigue tests[4,49] are strength tests that are conducted in an active environment over a wide range of loading rates to quantify parameters that describe slow crack growth. Setting aside for the moment issues of environment, loading rate, and natural flaw distribution versus indentation flaws, all of which are addressed below, the mechanics associated with measuring strength are the same for inert strength, Weibull distribution, or dynamic fatigue.

Bend Tests. Strength tests are usually conducted under uniaxial[60–63] or biaxial[64–68] bending conditions. The stress states associated with both of these bending geometries are well known, so data analysis is straightforward. A brief discussion of the advantages and disadvantages of each of these stress tests follows.

The most commonly used uniaxial bend test is called the 4-point bend test. This test, which is thoroughly described in the ASTM standard C 1161-94,[63] consists of a rectangular parallelepiped specimen in a loading fixture that consists of two parallel cylindrical loading pins in contact with one face of the specimen and two more widely spaced cylindrical loading pins in contact with the opposite face. The loading pins are perpendicular to and are spaced symmetrically about the center point of the specimen's long axis. This loading geometry has the advantage of generating a uniform, uniaxial stress longitudinally on the tensile side of the bend bar; the size of the uniform tensile region matches the spacing of the narrowly spaced loading cylinders. The large region of uniform tensile stress makes the 4-point bend geometry much preferable to the 3-point bend geometry, also described in ASTM standard C 1161-94.[63] In the 3-point bend geometry, the tensile stress is largest at the center of the specimen but drops off immediately as a function of distance away from the center. Therefore, in the 4-point bend geometry, a much larger population of initial defects is sampled at maximum stress than is sampled in the 3-point bend geometry.

Edge failures pose a problem in uniaxial testing. The process of machining specimen material into bars unavoidably generates machining damage along the edges of the specimens. If the stress intensity at one of the machining flaws is greater than the stress intensity values at the flaws of interest, the specimen will fail from the machining flaw. This gives rise to a couple of difficulties. On the simplest level, if specimens that fail from the edge are ignored in the analysis, edge failures still involve time and material that are wasted. On a more subtle level, the question arises whether it is legitimate to ignore data from specimens that fail from the edge? How can it be determined whether a specimen failed from a flaw that resulted from specimen manufacture or whether it failed from a flaw of interest that happened to lie near or at an edge? Certainly, in the latter case, discarding the data is not appropriate. Clearly, it would be better to avoid edge failures altogether. Two ways of accomplishing this for uniaxial bending involve either machine chamfering or hand rounding specimen edges on the tensile surface or use of a standard flaw large enough to be the source of failure. Edge modification requires that the 90° corners along the longitudinal tensile edges of the specimen be machined away or rounded the length of the specimen. This approach greatly reduces the probability of edge failures by eliminating the high stress concentrating effect of the corner. However, it reduces the tensile surface area of the specimen and adds both machining expense and time to the specimen preparation. In addition, a sloppy chamfering or rounding job can cause more damage than it relieves. Nevertheless, if carefully done, chamfering or hand rounding corners can nearly eliminate edge failures. The second approach to reduce edge failures through the use of large standard sized flaws will be discussed below for both uniaxial and biaxial bend tests. At this point, it is sufficient to say that the use of standard flaws clearly is not a solution to edge failures if the purpose of the strength tests is to obtain a Weibull distribution (see Appendix 1). However, standard flaws can be very useful in dynamic fatigue experiments.

Biaxial tests generate an equi-biaxial stress state on the tensile surface of disk-shaped specimens.[64–68] This is accomplished through the use of a test geometry in which a ring composed of small balls* (e.g., tungsten carbide balls) is placed in contact with one side of the specimen. This ring has a radius somewhat smaller than the radius of the specimen disk. On the other side of the disk, a ring or circular flat (e.g., a tungsten carbide ball that has had a flat machined onto it) is pressed against the specimen. When the flat is pressed against the specimen, an equi-biaxial tensile stress with a radius equal to the radius of the small flat is generated on the tensile surface of the specimen. The literature contains descriptions of biaxial test configurations in which a ball, rather than a flat or ring, is used to apply the load.[69] Like the 3-point bend test, point loading under biaxial conditions generates tensile stresses that fall monotonically from the center of the specimen; there is no region of high uniform tensile stress like that which occurs when a flat is used. Therefore, flat-on-ring rather than ball-on-ring loading is recommended.

Biaxial loading has several major advantages. First, because the stress is equi-biaxial, the most severe flaw will cause failure regardless of its orientation in the specimen. Second, because the loading geometry is circularly symmetric, alignment in the plane of the disk is less of a problem than for uniaxial bend tests. Third, because the high stress region is in the center of the disk and the stress at the edge of the specimen is only about 30% of the maximum stress, edge failures are almost completely eliminated. However, these benefits can be offset by the fact that biaxial testing requires much more material that uniaxial testing.

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*A solid ring of a somewhat compliant material may also be used.[22,68]

Both uniaxial and biaxial bend tests are subject to errors resulting from loading train misalignment and friction. The ASTM standards address both of these issues. Here we only mention the existence of the problems and commend the standards to the attention of the reader.

Standard Flaws. If it is not necessary to measure strengths from flaws representative of the flaw population in the final components, i.e., to determine the Weibull distribution, it is usually beneficial to generate a large, uniform flaw from which the specimen will fail. Such flaws can greatly reduce scatter in the strength data, allowing trends to be observed that might be obscured by the scatter resulting from the natural flaw population. Typically, such flaws are generated by use of a Vickers indenter to place an indentation impression in the center of what will be the high tensile stress region of the specimen. When indentations are used, their depths should remain fairly shallow, since the tensile stress in the bend test decreases with depth into the specimen. A rule of thumb is that the indentation impression, including cracks, should be less than one-fourth the thickness of the specimen. Examining the fracture surface of a bend specimen after testing can verify this.

It is important to be aware that indentations leave a residual stress field around the indentation impression. Therefore, strength tests on indented specimens must take into account both the applied far-field stresses as well as the local residual stresses at the indentation. In the dynamic fatigue measurements, the residual stress field results in an expression usually expressed in terms of $N' = (3N + 2)/4$ rather than N (see Eq. 2).[6] A residual field also typically surrounds natural flaws.[53] The residual stress associated with point flaws is identical in form to that around an indentation. Even many surface scratches can be treated as a series of point flaws, due to the nonuniform crack formation that occurs along the length of the scratch.

Dynamic Fatigue Measurements. As stated at the beginning of this appendix, dynamic fatigue tests are simply strength tests that have been made in the environment of interest as a function of stressing rate. Therefore, the discussion regarding uniaxial and biaxial strength tests applies directly to dynamic fatigue tests. The purpose of dynamic fatigue measurements is to quantify crack growth parameters. For the power law crack growth expression, it is particularly necessary to determine N. As mentioned above, dynamic fatigue experiments are usually conducted on indented specimens to minimize scatter in the strength. From Eq. 2, we see that evaluating N' requires that the independent axis be plotted as $\log(\dot{\sigma})$. For even moderate values of N, the slope of the dynamic fatigue curve will be shallow (e.g., for $N = 30$, $1/(N' - 1) \approx 0.05$). Consequently, to obtain accurate values for N', it is necessary to take the dynamic fatigue data over as wide a stressing rate as possible. We recommend taking the data over five orders of magnitude in stressing rate, if possible. Data taken over less than three orders of magnitude should not be trusted. An analysis of the confidence with which N' (or N) is known (see Appendix 3) will give guidance as to whether the stressing rate range was large enough.

It should be mentioned that recent work suggests that dynamic fatigue measurements made at too high a velocity could give values for N that are somewhat too low.[70,71] The reported work reflects ongoing research, and a discussion of it lies outside the scope of this chapter; the upper velocity at which this effect occurs is dependent upon both material and environment. In addition, it is not clear that the variation in N is generally large enough to detect experimentally. At any rate, any proposed error in N that would result from measuring dynamic fatigue at too high a stressing rate would result in a conservative lifetime prediction. Therefore, at this time, we recommend as wide a stressing rate range as possible.

Indented Inert Strength. Indented inert strength measurements are made with indented specimens rather than specimens that will fail from their natural flaw population. As stated earlier in this appendix, inert strength measurements need to be made at a high loading rate in an inert (e.g., N_2 gas) environment. It is worth mentioning that regular dry nitrogen gas is sufficient; there is no measurable advantage in going to a super dry gas as long as the loading rate is high.

Best Practices

- For either uniaxial or biaxial bend tests, articulated, self-aligning fixtures should be used to minimize extraneous stresses (see ASTM Standard C 1161-94 for uniaxial testing[62,63] and Refs 64, 65, 67, 68 for a discussion of biaxial testing).
- Inert strength tests require high loading rate and inert environment. Enclosing the test rig in a glove bag and flowing N_2 gas through the bag is a simple way to achieve an inert environment.
- To minimize edge failures in bend bars, the edges can be rounded or chamfered.
- Vickers indentations are useful in reducing scatter in bend tests. In uniaxial tests, the indentation should be oriented so that one set of diagonal cracks will be perpendicular to the stress axis.
- Dynamic fatigue tests should be made over as wide a range of stressing rates as possible. Three orders of magnitude is the minimum range that should be considered.

Dangers

- 3-point bend or ball-on-circle tests should be avoided in favor of 4-point bend or flat-on-circle tests.
- Do not use indentations for Weibull strength tests.
- In biaxial tests, do not use a rigid punch as the flat.

APPENDIX 3: CONFIDENCE LIMITS

The lifetime analysis presented above is based on three sets of experimental data: (1) dynamic fatigue data for indented specimens, (2) inert strength data for indented specimens, and (3) inert strength distribution for the strength-limiting flaws (Weibull data). To estimate the confidence in a lifetime prediction based on Eq. (3) and these three data sets, i.e., the lifetime at a particular *level of confidence,* we need to determine the distribution of lifetimes that results from the experimental uncertainties in these data sets. The *nonparametric bootstrap technique*[21,22,50–52] is ideal for this purpose. The nonparametric bootstrap technique involves the creation of a collection of simulated data sets based upon the original data. If the original data set contained n data points, the simulated data sets also contain n values and are generated by the random selection of values from the original data set. After each value is selected to provide a simulated data point, it is returned to the original data pool so that every value selected for the simulated sets is selected out of the full collection of n values (i.e., a select and replace procedure). The new set of data is called a *bootstrap sample*. This procedure is repeated a large number of times and the statistic of interest is determined.

The requirement for the original data set is that the data be *independent and identically distributed* (iid). Clearly, if the data for the Weibull distribution and the indented inert strength are taken properly, they meet this criterion. Equally clearly, the dynamic fatigue data do not; the strength values depend upon the stressing rate. To use the bootstrap technique to simulate dynamic fatigue data, the following steps are taken. First, a linear regression analysis is performed on the dynamic fatigue data, regressing the logarithm of the fracture stress versus the logarithm of the stressing rate. The residuals of this regression analysis, namely, the failure stress minus the best-fit value, do meet the iid requirement. Therefore, for each loading rate, residuals are chosen using the select and replace procedure previously described. A fatigue stress is calculated from the selected residual and the fitted line and associated with the loading rate to simulate a dynamic fatigue data set. A new estimate of lifetime can be made from each simulated set of Weibull, indented inert strength and dynamic fatigue data. This procedure can be repeated thousands of times to generate a distribution of lifetimes. The value of the lifetime at any arbitrary confidence level, $y\%$ (e.g., 99%), is the lifetime such that $y\%$ of the lifetime estimates are greater than this value.

Best Practice

- The bootstrap technique should be used to evaluate the confidence limits for the parameters from *each* of the three data sets as well as the confidence limits for the overall lifetime. This can alert the user to problems in a particular experimental procedure (e.g., a wide enough loading rate range in the dynamic fatigue experiment).

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PART 6

MANUFACTURING

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CHAPTER 29

INTERACTION OF MATERIALS SELECTION, DESIGN, AND MANUFACTURING PROCESSES

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1 MANUFACTURING OBJECTIVE

Manufacturing operations are targeted toward the production of products or components that will adequately perform their intended task. Implicit in this statement is the generation of parts with the required geometrical shape and precision. Further, these parts must be made from selected materials, with internal structures and companion properties that have been optimized for the specific service environment that the product must withstand. The *ideal product* is one that is "just good enough." Anything better will usually incur added cost through higher-grade materials, enhanced processing, or improved properties that may not be necessary. Anything worse, and we may encounter product failure, dissatisfied customers, and possible unemployment.

As shown in Fig. 1, nearly every manufactured item goes through a series of activities that include elements of design, material selection, process selection, manufacture, evaluation (and possible failure analysis) and feedback. Along the way, multiple alternatives are considered, and a number of decisions must be made. Many of these decisions are judgmental in nature, not black and white, and the consequences can be highly interrelated.

Consider the following assignment. Send a group of engineering students to a local discount department store. Ask them to identify the specific toaster that

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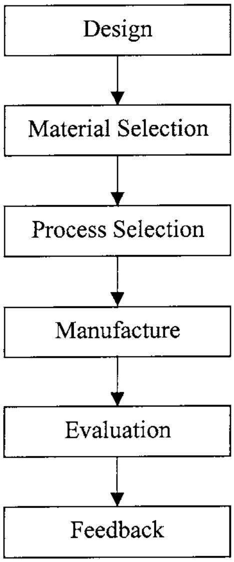

Fig. 1 Typical sequence of manufacturing activity.

they feel would be the best purchase for a college student and then prepare a brief justification of their recommendation. Invariably, some will find the two-slice model to be adequate, while others favor the four-slice feature. Most would select the thin-slot design, but a few would feel that thick bagels are a morning necessity that must be accommodated. Invariably there will be a range of qualities (good, better, and best) with companion increments in price. Are the more expensive models clearly better and best? Is the additional cost increment justified to upgrade from good to better, or yet another additional amount to go for best? Different students will select and defend different models. It is almost impossible to get a group consensus in view of the multiple judgmental factors that can be considered, and so it is with many of the decisions in manufacturing.

2 VARIOUS APPROACHES

There are several possible approaches that may be taken in the manufacture of products or components. One of the simplest is the case history approach. By accessing what has been done in the past (or done by a competitor), we can learn from that experience, use it as a starting base, and either duplicate or modify the details of that solution. A basic assumption of this approach is that similar requirements can be met with similar solutions.

While it is true that similar products can often be made with similar materials and processes, the real question is, How similar is similar? A minor variation in a service requirement (such as operating temperature or corrosive environment) may be sufficient to justify a totally different material or manufacturing method. In addition, this approach often overlooks new materials, new technology, and manufacturing advances that may have occurred since the formulation of the original solution.

It is also quite possible that the exercise is driven by aspects of current production. Evaluation of one of your products may have revealed some form of deficiency. A major customer may have requested a product just like the one you are currently making, but capable of operating at higher temperature, or in an acidic environment, or at higher pressure. With access to all of the details of the current part and its present method of manufacture, you must now determine how the problem should be overcome or the additional requirement accommodated.

The most comprehensive approach to part manufacture would be for an entirely new product where there is no existing reference material or process. Here the process begins by defining, in a very precise way, what it is that we want to produce, how it will be used, what conditions it will see, and what properties it must possess. The answers to these and related questions form a set of requirements that will then enable the evaluation of candidate materials and companion methods of fabrication. For completeness, it is this approach that will be further developed.

3 DESIGN

The first step in any manufacturing problem is to define the needs of the product, i.e., to describe the "target" in complete detail. The word *design* is often used to describe this process and entails the specification of geometric requirements, property requirements, and manufacturing requirements for each component of the assembly. It is the responsibility of the designer to identify the specific properties or characteristics that will be necessary for each part to adequately perform its intended function.

A dimensioned sketch can begin to answer many of the questions about the size, shape and complexity of each part. Additional details, however, are often essential. How precise must the dimensions be? Are all dimensions precise or only a certain fraction, and, if only a few, which ones? What are the surface finish requirements? If all surfaces do not have the same requirements, which ones are restrictive and what fraction do they comprise? Are there axes or planes of symmetry? Does the part have a uniform cross section when viewed from a certain direction? How does this component interact geometrically with the other components?

While the above questions may be viewed as simply defining *geometrical needs,* they will actually form the basis for subsequent decisions. At some point, for example, we will have to determine how to manufacture the requested shape. We know that die casting, for example, can be used for parts that range from less than an ounce to more than 100 lb, but wall thicknesses should ideally be less than $\frac{5}{16}$-in. Permanent mold casting extends the wall thickness limit to 2 inches, and there is no limit at all for sand casting. At the same time, dimensional precision and the as-cast surface finish becomes progressively worse as we move from die casting to permanent mold casting to sand casting. Secondary operations, such as machining, grinding, or polishing, all involve the handling, positioning, and processing of individual parts and can form a significant portion of the total manufacturing cost. Therefore, the objective is usually to "hit the target" with the least number of operations. Extrusion and rolling are metal forming processes that are known for their ability to produce long pieces of constant cross section. Powder metallurgy products must have shapes that can be ejected from a die. Thus, we may quickly have some specific processes in mind, and may want to consult the designer if it appears that a possible geometric modification would significantly improve either the ease of manufacture or subsequent performance of the part.

Getting the right shape is only part of the desired objective. If a part is to perform adequately, it is imperative that it also possesses the necessary mechanical and physical properties, as well as the ability to endure the anticipated environments for a desired period of time. Consideration and specification of these properties is another aspect of component design.

Let us begin by considering some of the possible *mechanical property requirements*. Will the part need a certain level of static strength? If it is accidentally overloaded, is it permissible to have a sudden brittle failure or is deformation or distortion a desirable precursor to fracture? How much can the component bend or deflect under load and still function properly? Are impact loadings expected? If so, what will be the type, magnitude, and velocity? Are vibrations or cyclic loadings anticipated? Will they be uniform or varying? What is the anticipated frequency and magnitude? Is wear resistance desired? Where and how deep? Will all of the above requirements be necessary over the entire range of operating temperatures?

Not all of the requirements will be mechanical. Some may be electrical, magnetic, thermal, or optical. These *physical property requirements* must also be considered. Must the component be an electrical insulator or conductor? Are any magnetic properties desirable? Is there a need for thermal conductivity? If temperature will change during operation, must thermal expansion or contraction be restricted or matched to other components? Are there any optical requirements? Is weight a factor? What about overall appearance or the availability in specified or varied colors?

Another area of consideration involves the interaction of the component with its operating environment, that is, *environmental concerns*. What are the lowest, highest, and normal operating temperatures? How fast might temperature change? Are the temperature changes cyclic or varied? Are all of the above properties required over the entire range of temperatures? What is the most severe operating environment from the perspective of corrosion or deterioration of material properties? What is the desired lifetime of the product? Should it be manufactured for ease of repair, disposal, or recycling? What is the potential liability if the product should fail?

The above considerations are only a sample of the many questions that must be addressed when precisely defining what it is that we want to produce. Collectively, they will serve to direct and restrict the selection of engineering material. It is possible that the requirements will be such that several families of materials, and numerous members of those families, will all be adequate, and selection becomes a matter of preference. It is also possible that one or more of the requirements will emerge as a dominant restriction (such as the need for ultrahigh strength, superior wear resistance, the ability to function at high operating temperatures, or the ability to withstand extremely corrosive environments), and selection becomes focused on those materials offering that unique characteristic.

Help in identifying candidate materials or families of materials may be found by classifying the various requirements as ‘‘absolute’’ or ‘‘compromisable.’’ Absolute requirements are ones that must be met or the part will fail. Materials that fall short of absolute requirements are automatically eliminated. Compromisable properties are those that might differentiate good, better, and best, where all would be deemed adequate. These requirements are helpful in prioritizing materials where more than one candidate emerges.

Still another family of factors remains to be specified. We might call these *manufacturing concerns* because they will ultimately be used to help select an economical manufacturing process or processes. Foremost among these factors might be the number of identical parts to be produced and the desired rate of production or anticipated rate of consumption. One-of-a-kind parts are rarely made by processes that require dedicated patterns, molds, or dies. The expense of dedicated tooling is too hard to justify for a production run of one. Instead, flexible processes, such as machining, are likely to be used where the skill of an operator generates the unique details of the part. Processes that only produce one part an hour may be quite adequate for aerospace assembly where the production rate is only several planes per month. In contrast, processes that require more than several seconds per part may be unattractive for automotive components that will be used on high-volume models.

Additional manufacturing concerns include the desired level of quality (compared to similar products on the market) and the related quality control and inspection requirements. It may be necessary, for example, to provide inspection access to specific surfaces or locations on a product, and this requirement would need to be addressed as the manufacturing process and sequence of activities is formulated. Assembly (or disassembly) concerns and key relationships between mating parts also need to be noted.

As we progress through the design stage, it is important to define the entire spectrum of needs. The focus should be on the various ‘‘requirements,’’ and we should avoid jumping to ‘‘solutions.’’ Nevertheless, the need or desirability for certain manufacturing processes may become blatantly obvious. If this is so, we might also want to note the desirability for material characteristics such as enhanced machinability, weldability, formability, castability, or hardenability.

Time spent in defining the specific needs or requirements of a product is indeed time well spent. It will enable reasonable decisions to be made, and will help to prevent costly errors or product failures that might result from some form of oversight.

Figure 1 depicted a series of activities moving from design to material selection and then to process selection. Figure 2 shows a modified form of Fig. 1 where the material selection and process selection activities are now parallel and not sequential. This figure reflects the fact that it is not uncommon for one of these two selections to assume a dominant role and the other to become secondary or dependent. For example, the production of a large quantity of a small, intricate part with thin walls, precise dimensions, and smooth surfaces may be an ideal candidate for die casting. If this process is clearly the most attractive

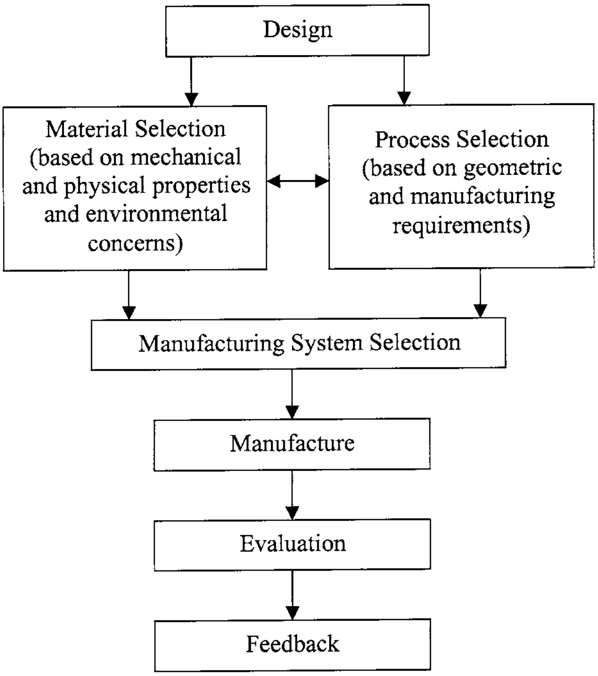

Fig. 2 Modified sequence of manufacturing activity showing parallel selection of material and process.

means of manufacture, material selection may be restricted to die-castable materials—assuming feasible alternatives exist. In a converse example, highly restrictive material properties, such as the ability to endure extreme elevated temperatures or severe corrosive environments, may significantly limit the material options. The fabrication options will be focused on those that are compatible with the candidate material. Figure 3 schematically presents some of the commonly accepted material—process compatibilities and incompatibilities.

4 MATERIAL SELECTION

A wide spectrum of engineering materials is currently available, each with its own unique set of strengths and limitations. It is possible that one material will emerge as the obvious choice for meeting the design requirements. More likely, there will be multiple candidates that will each meet the requirements to varying degrees. Selection now becomes a matter of preference with the final choice reflecting the relative merits of the entire manufacturing systems (material and complete method of fabrication). It may also be possible that no existing material is capable of meeting the stated requirements, or the materials that are feasible are eliminated because of features such as high cost, limited availability, environmental concerns, or the need for recyclability. In these cases, alternatives must be made possible through redesign, compromise of requirements, or the development of new engineering materials.

Material Process	Irons	Steel	Aluminum	Copper	Magnesium	Nickel	Refractory Metals	Titanium	Zinc
Sand Casting	X	X	X	X	X	X			O
Permanent Mold Cast	X	O	X	O	X	O			O
Die Casting			X	O	X				X
Investment Casting		X	X	X	O	O			
Closed-die Forging		X	O	O	O	O	O	O	
Extrusion		O	X	X	X	O	O	O	
Cold Heading		X	X	X		O			
Stamping, Deep Draw		X	X	X	O	X		O	O
Screw Machine	O	X	X	X	O	X	O	O	O
Powder Metallurgy	X	X	O	X		O	X	O	

Key:

X = Routinely performed
O = Performed with difficulty, caution, or some sacrifice (such as die life)
Blank = Not recommended

Fig. 3 Commonly accepted material-process compatibilities.

It should also be noted that it is not uncommon to have to make decisions with incomplete information. Not all of the desired data may be available, or the data may be for test conditions that differ somewhat from those anticipated for the part in question. This is yet another example of where educated judgment is required.

5 SELECTION OF FABRICATION PROCESS

Our objective is to produce parts with the right shape and companion properties that will enable successful performance of the intended function. We must now consider the various ways of producing the desired shape and evaluate each with regard to its compatibility with the candidate materials.

The possibilities here are numerous. *Material removal processes* start with an oversized piece of material and then remove some so as to leave the desired shape. All of the traditional machining processes are included here: turning, drilling, milling, boring, and others. Also included in this category are grinding, electrodischarge machining (EDM), electrochemical machining (ECM), waterjet cutting, and other nontraditional processes.

A number of *casting processes* have been developed based on the premise that a liquid assumes the shape of its container. By producing shaped molds, and solidifying the material in the mold, a wide variety of extremely complex shapes can be produced in an equally wide range of sizes.

Deformation processes exploit the plasticity of engineering materials, utilizing applied forces to induce movement or flow. Rolling, extrusion, forging, wire drawing, sheet metal forming, and other processes can be used to mass produce parts from ductile materials.

An equally diverse group of processes can be used to produce shapes by joining smaller pieces together. These *consolidation processes* include welding, brazing, soldering, industrial adhesives, mechanical fasteners, and powder metallurgy.

Selection of one or more candidate processes begins with an evaluation of the previously discussed geometric requirements and manufacturing concerns. Key geometric features include size, shape, complexity, and symmetry, as well as the desired dimensional precision and surface finish. Dominant manufacturing concerns include the desired quantity and rate of production.

With a clear picture of the requirements and desires, we can begin to evaluate the alternatives. Each of the manufacturing processes has distinct advantages and limitations, and these can often form the basis for a prescreening. Further refinement can be based on a number of considerations. Is the process compatible with candidate materials? Is the size of the part, including features such as wall thickness, consistent with process capabilities? If the process is used, how precise will the final dimensions be? What is the expected surface finish? Does the process take advantage of any product symmetries or other geometrical features? What is the form of the required starting material and are there any associated economies that should be noted? Will the process require special tooling (such as molds, dies, fixtures, etc.) that will be unique to the desired part, and can the cost be justified?

For each of the possible materials, one or more compatible processes (or series of processes) should be identified, resulting in candidate *manufacturing systems*. At this stage, each system consists of a material and companion fabrication process. By comparing their relative merits, it is hoped that we can clearly identify a “best” alternative.

6 COMPLETING THE SYSTEM: SECONDARY PROCESSING

It is rare for a single manufacturing process to produce a part with both the desired shape and the desired properties. One process may produce the desired dimensional precision in a single operation, while another less expensive alternative might require some secondary machining. Grinding or polishing may be necessary to produce surfaces with the desired surface finish. These secondary processes must be incorporated into each of the candidate manufacturing systems. Moreover, because these secondary processes incur the added expenses of handling, positioning, fixturing, and associated tooling, it is not uncommon for them to be among the most expensive stages of the system.

In addition to meeting the geometric requirements, we must also assure the necessary properties. While each of the candidate materials was selected for its ability to possess the desired properties, these properties may not be present when the part emerges from the shape-producing process. Secondary processing may be required to induce property modification. Heat treatments, for example, can significantly alter the mechanical properties of engineering metals and other materials. Surface treatments can provide enhanced wear resistance, enhanced corrosion resistance, or the appearance features that will promote sales and marketability.

7 SELECTION OF "BEST" SYSTEM

We began with well-defined needs and objectives and proceeded to identify candidate manufacturing systems, each of which begins with a starting material and includes everything that must be done to convert it into the desired final product. Every one of the options involves a series of highly interrelated decisions. Performance and property needs restrict candidate materials. Various materials limit the fabrication possibilities. The fabrication method, in turn, affects the material properties. Processes designed to modify certain properties may simultaneously alter other properties.

Selection of the best alternative requires a complete understanding of all of the various restrictions and interrelations incorporated in each candidate system. In addition to a performance evaluation, consideration should also be given to the costs associated with both material and processing, environmental safety, recycling or reclamation of both the end product and processing wastes, minimization of required energy, and aspects of process control, quality control of the product, and product liability.

Because of the need to maintain multiple perspectives and incorporate diverse expertise, it is not uncommon for manufacturing decisions to be made by a team of individuals. Representation from the design function is essential to assure that each of the various requirements is met and that any adjustment in those requirements is acceptable. Materials specialists bring expertise in candidate materials, their structure and properties, and the effects of various processing. Manufacturing personnel know about the capabilities of various processes, as well as the equipment available and the cost of associated tooling. Quality and environmental specialists bring their perspective and expertise. Failure analysis personnel can provide valuable experience gained from their examination of unsuccessful efforts. Customer representatives or marketing specialists may also be consulted for their opinions. Clear and open communication is vital to the making of sound decisions and compromises.

The design and manufacture of a successful product is an iterative and evolving process. Costs change, prototype or customer assessment may reveal unexpected weaknesses or the possibility of improvements, and manufacturing problems may be encountered. A change in material may well require a change in manufacturing process, and conversely improvements in processing may warrant a reevaluation of material. It is vitally important to maintain an understanding of the various system interactions to assure that any change or corrective action will not adversely affect other aspects of the system.

8 INTERRELATIONSHIP EXAMPLES

To illustrate some of the many interrelationships between design requirements, engineering materials, and manufacturing processes, let us now consider a series of examples:

Example 1: Products Made from Crystalline Ceramics

Crystalline ceramic materials are ionic or covalently bonded and therefore exhibit a characteristic brittleness. The absence of ductility makes the plastic deformation processes unavailable to these materials. High bond strengths impart the characteristic of high melting points. Casting processes require the handling of molten materials, and the high melting temperatures present considerable problems. In addition, nonuniform cooling from high temperatures can generate high residual stresses, often sufficient to induce fracture in the product. As a result, casting processes are generally not considered for the crystalline ceramics. For similar reasons, joining processes that involve molten material, such as fusion welding, are likewise eliminated. Mechanical fasteners lead to concentrated stresses and again the likelihood of brittle fracture. Therefore, joining is largely limited to adhesive or diffusion bonding. Many of the material removal processes involve some form of mechanical cutting action, and this also would result in fracture of ceramic materials. Material removal, therefore, is usually restricted to grinding, polishing, or chemical etching. Having eliminated a large number of fabrication processes as being incompatible with ceramic materials, we find that the production of a desired shape is often limited to processes that bond together a multitude of small particles, i.e., particulate processing. The design and manufacture of ceramic products, therefore, is frequently restricted by the limitations of particulate processing.

Example 2: Classroom Chalk Tray

As another example of the interrelation between design, materials, and processing, consider the common classroom chalk tray. The requirements here are not very restrictive. Depending upon design, the chalk tray may be required to support the static weight of the writing board, and most will sometime encounter the leaning weight of a tired or casual instructor. Otherwise, the demands are quite minimal. Wood has been a longtime standard for this application and usually employs adhesive bonding to produce the necessary lengths, coupled with some form of machining operation designed to produce the long, continuous cross-section products. Some negative concerns are the role of grain and knots in fracture and splintering, as well as the need to provide periodic maintenance in the form of surface refinishing. As a competing material, aluminum sections offer light weight, enhanced durability, and low maintenance. Since aluminum is highly ductile, the extrusion process can be used to produce the desired cross section in the necessary lengths. Extruded thermoplastic polymers offer yet another competitive system with lower strength and durability, but also lower cost.

Example 3: Household Window Frames

The continuous-cross-section components of a household window frame bear considerable similarity to our classroom chalk tray, but bring some additional requirements. The appearance of both external and internal surfaces is quite important, as well as the desire for low maintenance. Since the pieces provide a continuous transition from interior to exterior, thermal conductivity (i.e., insulating ability) is yet another concern. Wood sections are quite attractive and can utilize a wide spectrum of finishes and coatings. As with the chalk tray, however, there are concerns relating to grain, knots, and the required periodic maintenance of repainting or refinishing. The possibilities of swelling, shrinking, and warping are additional concerns for the window application. Aluminum has all of the advantages described with the chalk tray, plus the ability to be inexpensively finished through color anodizing to produce a range of desirable colors and textures. The high thermal conductivity is a distinct disadvantage that may lead to sweating or condensation in winter, but this may be overcome through a design that incorporates some form of thermal barrier. Extruded vinyl will probably meet all of the mechanical requirements. The attractive feature of integral color (obtained by incorporating dyes or pigments with the resin) eliminates the need for both internal and external finishing operations. Dimensional instability over time, low-temperature brittleness, and the deteriorating effects of ultraviolet light are some of the negative concerns.

A survey of building supply warehouses or actual homes will reveal that each of the above materials currently commands a segment of the market. There is no clear-cut winner, and selection becomes largely a matter of pros, cons, and preferences.

Example 4: Bamboo

Material science education has long recognized the interrelation between structure and properties, and this relationship forms a major portion of most introductory courses. As an example of such a relationship, consider the natural material known as bamboo. Bamboo is a long, segmented, tubular material composed of longitudinal fibers bonded by a weaker lignin matrix. Longitudinal fractures are somewhat easy to form in this material, since they require only the rupture of the weaker lignin to separate the parallel fibers. Transverse fracture, however, requires rupture of the stronger fibers. Moreover, since the fibers are independent of one another, the fracture does not propagate continuously. Instead, each fiber must break individually, producing the characteristic ragged fracture. A transverse fracture, therefore, requires far more energy than a longitudinal one. Now let us extrapolate this observation to a far more critical application.

Example 5: Aerospace Turbine Blades

The individual turbine blades used in the exhaust regions of jet engines must withstand high temperatures, high stresses, and highly corrosive operating conditions. Because of the life-threatening consequences, failure by fracture is totally unacceptable.

The extremely demanding operating requirements severely limit the material possibilities, and most jet engine turbine blades have been fabricated from one of the high-temperature superalloy metals. Fabrication processes are limited to those that are compatible with both the material and the desired geometry. Through the 1960s and into the early 1970s, investment casting was the standard method of fabrication. This resulted in a polycrystalline product with thousands of spherical or polygonal crystals. Because of the high operating temperatures and high centrifugal forces, the failures that did occur usually assumed the form of transverse fracture at the base of the blades, with the fracture path following the weaker intercrystalline boundaries.

In the 1970s, a fabrication advance enabled turbine blade production via unidirectional solidification. The product was still polycrystalline, but there were fewer crystals and they ran the entire length of the blade. The intercrystalline surfaces of weakness all ran longitudinally, perpendicular to the direction of likely failure. In essence, the turbine blades now resembled bamboo. Transverse fracture was impaired and the "lifetime" of the turbine blades (use time before mandatory replacement) was significantly extended.

Recent manufacturing advances have now made single-crystal turbine blades an economical possibility. By eliminating all internal surfaces of weakness, performance and lifetime are further enhanced. Figure 4 shows a side-by-side comparison of the polycrystalline, unidirectionally solidified, and single-crystal structures.

This example clearly illustrates yet another important interaction feature. The structure–property aspect of material education has been expanded to one of processing–structure–properties–performance. While educators tend to emphasize structure and properties, industry tends to focus on processing and performance. This example, however, illustrates the complete interaction since processing advances enabled production of the altered structure. The associated change in properties was responsible for the enhanced performance.

Example 6: Forging and Oriented Grain Flow

According to the tenets of modern fracture mechanics, the role of any internal flaw or defect can vary significantly with both size and orientation. Flaws that

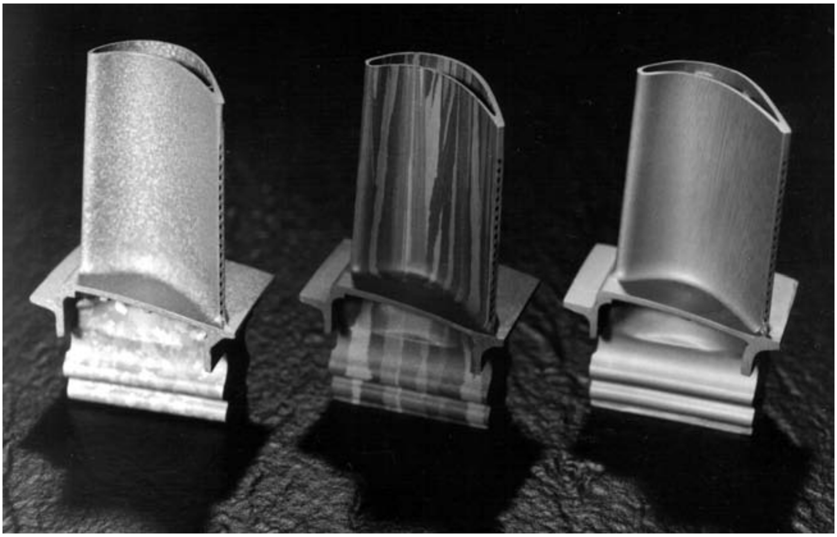

Fig. 4 Comparison of polycrystalline, directionally solidified, and single-crystal turbine blades. (Courtesy of Howmet Corporation, Darien, CT)

intersect free surfaces in a perpendicular or near perpendicular orientation are referred to as ‘‘crack initiators.’’ Subsurface flaws of similar orientation are ‘‘crack propagators,’’ and subsurface flaws that are aligned parallel to external surfaces are ‘‘crack arrestors.’’

Closed-die (or impression-die) forgings tend to be three dimensionally complex metal components that are used in mechanically demanding applications that are often prone to fatigue failure. One of the more common metal flaws or defects is the nonmetallic inclusion, and these inclusions are commonly found along intercrystalline boundaries.

Casting processes are common means of producing three dimensionally complex shapes. The resulting structure is typically a polycrystalline solid composed of equiaxed (spherically symmetrical) crystals. Since there is no preferred orientation of grain boundaries, there is an equal probability of both the damaging crack initiation or crack propagation orientation and the desirable crack arresting geometry.

Machining a starting block of material is another means of producing the desired final shape. The starting material, frequently a rolled plate or bar, has a prior processing history that affects its structure. Rolling operations elongate the crystals, as well as the flaws or defects that lie along grain boundaries, in the direction of rolling. Since the machining operation then cuts through the prior structure, any cut surface that crosses the rolling direction has a high probability of exposing crack initiation flaws or having underlying crack propagators.

Forging operations also begin with a solid piece of material that has typically been rolled in its prior processing. During the forging operation, however, the plastic deformation further reorients the grain structure and associated flaws or defects. Where the starting piece was sheared from a larger plate or bar, the sheared surfaces will resemble the machined surfaces discussed above, and flaws or defects can be detrimental. Surfaces created by the subsequent flow of the forging operation typically have the inclusions in a crack arrestor orientation. Thus, by orienting the flow of highly stressed surfaces, forged components can offer enhanced fracture resistance and fatigue life.

Example 7: Sheet Metal Blades in a Flex Fan

Internal combustion engines frequently require an associated cooling system that typically includes both internal water cooling and the external flow of cooling air. Large truck engines frequently employ a ‘‘flex-fan’’ to provide the external airflow. Curved sheet metal blades are riveted to a central rotating hub with spider arms. When the engine is at high rpm, the assumption is that the vehicle is in forward motion and the airflow provided by this motion is sufficient to provide the necessary cooling. Centrifugal force uncurls the blades, thereby reducing the aerodynamic drag of the rotating fan. When the engine drops to low rpm or idle, the vehicle is frequently at low speed or stand still and forward motion no longer provides a sufficient flow of air. The slow rotation speed permits high blade curvature and a large volume of displaced air.

During the manufacturing operation, each of the sheet metal blades is rigidly fixed along the edge where it is clamped to the rotating spider. The remainder of the blade is free to flex about this joint. When failure occurs, it is frequently by a fracture that propagates along the spider arm where the flexing blade encounters restraint. Key to enhanced performance is the orientation of the rolled sheet metal blade with respect to the spider arm. If the rolling direction of the blade is parallel to the spider arm, the inclusions that have been elongated and oriented in the rolling operation are all aligned as crack initiators or crack propagators. If the rolling direction were reoriented by 90°, the grain orientation and defect alignment is now perpendicular to the direction of likely failure. All of the flaws become crack arrestors and performance is enhanced.

Example 8: Repair Welds in Metal Products

The interrelation between processing, structure, properties, and performance for specific materials can be further illustrated by a consideration of repair welds. Fusion welding creates a pool of molten metal through the melting of both filler metal and base metal. This region of a weld is subject to all of the problems and difficulties of a metal casting, including as-solidified grain structure, gas absorption and evolution, and solidification shrinkage. Moreover, if both segments of a weld are restrained during the shrinkage, the weld becomes stressed in tension and cracking may occur in the weakest regions of the structure.

Adjacent material is subjected to heating and cooling during the welding process. Immediately adjacent to the weld pool, temperatures reach almost to the melting point. As we move further from the weld, the peak temperature drops. Within a certain region, known as the heat-affected zone, the exposure to elevated temperature is sufficient to alter both the structure and properties of the material. The resultant features depend not only on the peak temperature, but also on the rate of heating and cooling, which further depends upon the welding process and the geometry of the components. Fusion welding, therefore, can be viewed as a metal casting in a metal mold, coupled with a complex and highly varied heat treatment of the adjacent metal.

Consider a repair weld in a component that has been made from quenched-and-tempered heat-treated steel. A segment of the heat-affected zone immediately adjacent to the molten metal will attain temperatures well above the critical temperature necessary to reaustenitize the material. Upon cool-down to room temperature, this material will form an entirely new structure, which will vary, depending upon the rate of cooling. Further away from the weld, material will attain a temperature that is high enough to produce atomic diffusion, but not high enough to revert the structure to austenite. Here, the material will continue to temper, making the metal softer, weaker, and more ductile. The result is a newly deposited casting and an adjacent region with highly varied structure and a wide range of possible properties. Since most of the alterations are considered to be detrimental, considerable caution should be exercised when welding quenched-and-tempered heat-treated steels.

Consider a repair weld in age-hardened aluminum, such as 7075-T6, and again focus attention on the heat-affected region. Adjacent to the weld is a region that will experience temperatures above that required to redissolve the precipitate. Upon cooling, the material will try to produce a two-phase structure, with the final form and associated properties depending upon peak temperature, time at temperature, and cooling rate. Further from the weld will be a region of the heat-affected zone where atomic diffusion will further the aging process. Overaging leads to lowered strength and hardness, and this region may well become the site of future failure.

As a final example, consider a weld in metal that has been strengthened by cold working. Heating of this material above the temperature required for atomic diffusion will promote the processes of recovery, recrystallization, and grain growth. Associated with each are companion changes in structure, properties, and performance.

CHAPTER 30

PRODUCTION PROCESSES AND EQUIPMENT FOR METALS

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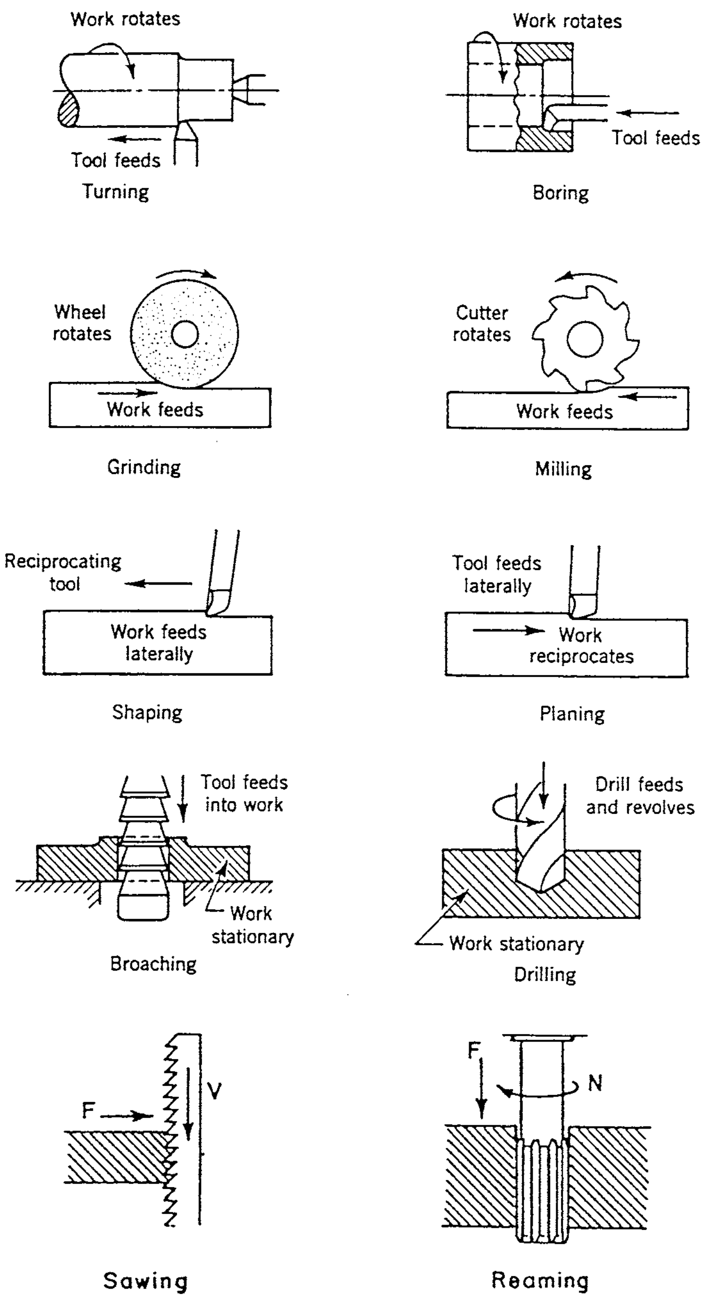
1 METAL-CUTTING PRINCIPLES

Material removal by chipping process began as early as 4000 BC, when the Egyptians used a rotating bowstring device to drill holes in stones. Scientific work developed starting about the mid-19th century. The basic chip-type machining operations are shown in Fig. 1.

Figure 2 shows a two-dimensional type of cutting in which the cutting edge is perpendicular to the cut. This is known as *orthogonal* cutting, as contrasted with the three-dimensional *oblique* cutting shown in Fig. 3. The main three cutting velocities are shown in Fig. 4. The metal-cutting factors are defined as follows:

α	rake angle
β	friction angle
γ	strain
λ	chip compression ratio, t_2/t_1
μ	coefficient of friction
ψ	tool angle
τ	shear stress
ϕ	shear angle
Ω	relief angle
A_o	cross section, wt_1
e_m	machine efficiency factor
f	feed rate ipr (in./revolution), ips (in./stroke), mm/rev (mm/revolution), or mm/stroke



Turning

Boring

Grinding

Milling

Shaping

Planing

Broaching

Drilling

Sawing

Reaming

Fig. 1 Conventional machining processes.

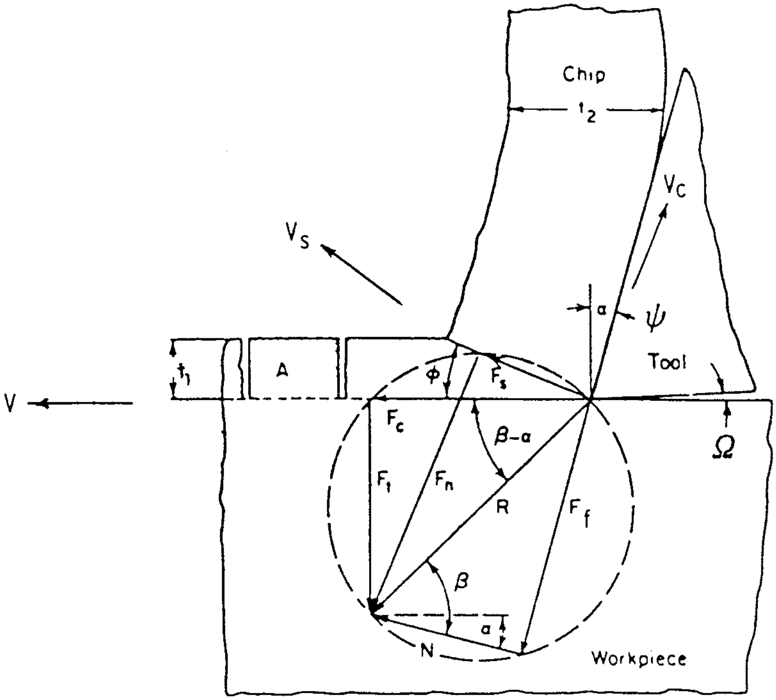


Fig. 2 Mechanics of metal-cutting process.

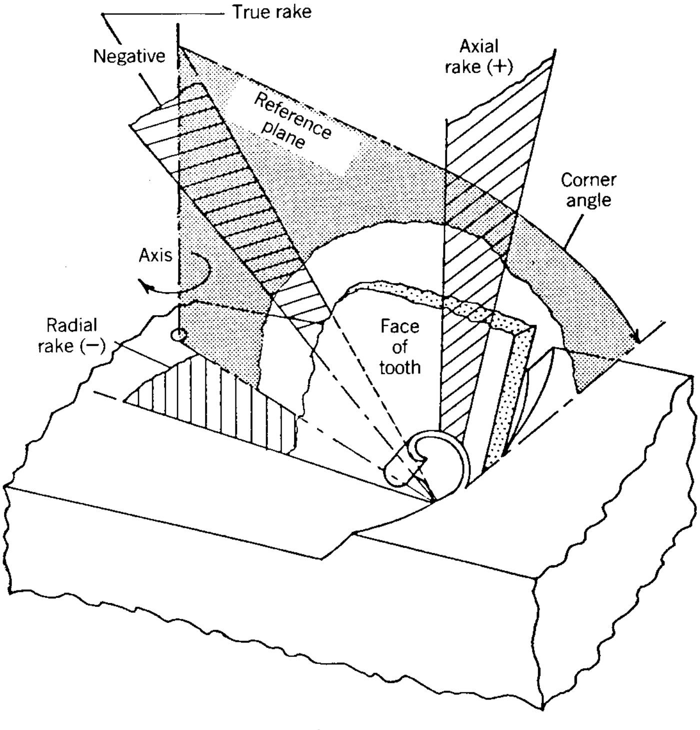


Fig. 3 Oblique cutting.

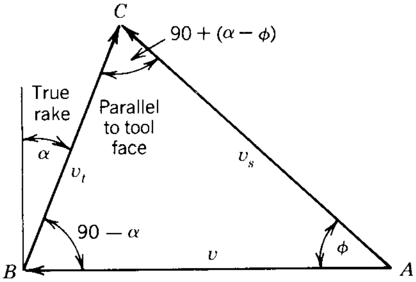


Fig. 4 Cutting velocities.

f_t	feed rate (in./tooth, mm/tooth) for milling and broaching
F	feed rate, in./min (mm/sec)
F_c	cutting force
F_f	friction force
F_n	normal force on shear plane
F_s	shear force
F_t	thrust force
HP_c	cutting horsepower
Hp_g	gross horsepower
Hp_{μ}	unit horsepower
N	revolutions per minute
Q	rate of metal removal, in.^{3}/min
R	resultant force
T	tool life in minutes
t_1	depth of cut
t_2	chip thickness
V	cutting speed, ft/min
V_c	chip velocity
V_s	shear velocity

The *shear angle* ϕ controls the thickness of the chip and is given by

$$\tan \phi = \frac{\cos \alpha}{\lambda - \sin \alpha} \tag{1}$$

The *strain* γ that the material undergoes in shearing is given by

$$\gamma = \cot \phi + \tan(\phi - \alpha) \tag{2}$$

The *coefficient of friction* μ on the face of the tool is

$$\mu = \frac{F_t + F_c \tan \alpha}{F_c - F_t \tan \alpha} \tag{3}$$

The *friction force* F_t along the tool is given by

$$F_t = F_t \cos \alpha + F_c \sin \alpha \tag{4}$$

Cutting forces are usually measured with dynamometers and/or wattmeters. The shear stress τ in the shear plane is

$$\tau = \frac{F_c \sin \phi \cos \phi - F_t \sin^2 \phi}{A} \tag{5}$$

The speed relationships are

$$\frac{V_c}{V} = \frac{\sin \phi}{\cos(\phi - \alpha)}$$

$$V_c = V/\lambda \tag{6}$$

2 MACHINING POWER AND CUTTING FORCES

Estimating the power required is useful when planning machining operations, optimizing existing ones, and specifying new machines. The power consumed in cutting is given by

$$\text{power} = F_c V \tag{7}$$

$$HP_c = \frac{F_c V}{33{,}000}$$

$$= Q\, HP_\mu$$

where F_c = cutting force, lb
V = cutting speed, ft per min = $\pi DN/12$ (*rotating operations*)
D = diameter, in.
N = revolutions per min
HP_μ = specific power required to cut a material at a rate of 1 cu in. per min
Q = material removal rate, cu in./min

For SI units,

$$\text{Power} = F_c V \quad \text{watts} \tag{8}$$

$$= QW \quad \text{watts} \tag{9}$$

where F_c = cutting force, newtons
V = m per sec = $2\pi RN$
W = specific power required to cut a material at a rate of 1 cu mm per sec
Q = material removal rate, cu mm per sec

The specific energies for different materials, using sharp tools, are given in Table 1.

$$\begin{aligned} \text{power} = F_c V &= F_c 2\pi RN \\ &= F_c R 2\pi N \\ &= M 2\pi N \qquad (10) \\ &= \frac{MN}{63{,}025} \quad HP \qquad (11) \end{aligned}$$

where M = torque, in.–lbf
N = revolutions per min

In SI units,

$$= \frac{MN}{9549} \quad KW \qquad (12)$$

where M = newton–meter
$HP/\text{cu in./min } 2.73 = ?\ W/(\text{cu mm/sec})$
$M = F_c R = \text{power}/2\pi N$
$F_c = M/R$

Table 1 Average Values of Energy per Unit Material Removal Rate

Material	Bhn	HP_c/in.^{3} per min	W/mm^3 per sec
Aluminum alloys	50–100	0.3	0.8
	100–150	0.4	1.1
Cast iron	125–190	0.5	1.6
	190–250	1.6	4.4
Carbon steels	150–200	1.1	3.0
	200–250	1.4	3.8
	250–350	1.6	4.4
Leaded steels	150–175	0.7	1.9
Alloy steels	180–250	1.6	4.4
	250–400	2.4	6.6
Stainless steels	135–275	1.5	4.1
Copper	125–140	1.0	2.7
Copper alloys	100–150	0.8	2.2
Leaded brass	60–120	0.7	1.9
Unleaded brass	50	1.0	2.7
Magnesium alloys	40–70	0.2	0.55
	70–160	0.4	1.1
Nickel alloys	100–350	2.0	5.5
Refractory alloys (Tantalum, Columbium, Molybdenum)	210–230	2.0	5.5
Tungsten	320	3.0	8.0
Titanium alloys	250–375	1.3	3.5

$$\text{gross power} = \text{cutting power}/e_m \tag{13}$$

The cutting horsepowers for different machining operations are given below.

For turning, planing, and shaping,

$$HP_c = (HP_\mu)12CWVfd \tag{14}$$

For milling,

$$HP_c = (HP_\mu)CWFwd \tag{15}$$

For drilling,

$$HP_c = (HP_\mu)CW(N)f\left(\frac{\pi D^2}{4}\right) \tag{16}$$

For broaching,

$$HP_c = (HP_\mu)12CWVn_cwd_t \tag{17}$$

where V = cutting speed, fpm
C = feed correction factor
f = feed, ipr (turning and drilling), ips (planing and shaping)
F = feed, ipm = $f \times N$
d = depth of cut, in.
d_t = maximum depth of cut per tooth, in.
n_c = number of teeth engaged in work
w = width of cut, in.
W = tool wear factor

Specific energy is affected by changes in feed rate. Table 2 gives feed correction factor (C). Cutting speed and depth of cut have no significant effect on power. Tool wear effect factor (W) is given in Table 3.

Table 2 Feed Correction (*C*)

Factors for Turning, Milling, Drilling, Planing, and Shaping		
Feed (ipr or ips)	mm/rev or mm/stroke	Factor
0.002	0.05	1.4
0.005	0.12	1.2
0.008	0.20	1.05
0.012	0.30	1.0
0.020	0.50	0.9
0.030	0.75	0.80
0.040	1.00	0.80
0.050	1.25	0.75

Table 3 Tool Wear Factors (*W*)

Type of Operations[a]	*W*
Turning	
Finish turning (light cuts)	1.10
Normal rough and semifinish turning	1.30
Extra-heavy-duty rough turning	1.60–2.00
Milling	
Slab milling	1.10
End milling	1.10
Light and medium face milling	1.10–1.25
Extra-heavy-duty face milling	1.30–1.60
Drilling	
Normal drilling	1.30
Drilling hard-to-machine materials and drilling with a very dull drill	1.50
Broaching	
Normal broaching	1.05–1.10
Heavy-duty surface broaching	1.20–1.30

[a] For all operations with sharp cutting tools.

The gross power is calculated by applying the overall efficiency factor (e_m).

3 TOOL LIFE

Tool life is a measure of the length of time a tool will cut satisfactorily, and may be measured in different ways. Tool wear, as in Fig. 5, is a measure of tool failure if it reaches a certain limit. These limits are usually 0.062 in. (1.58 mm) for high-speed tools and 0.030 in. (0.76 mm) for carbide tools. In some cases, the life is determined by surface finish deterioration and an increase in cutting forces. The cutting speed is the variable that has the greatest effect on tool life. The relationship between tool life and cutting speed is given by the Taylor equation.

$$VT^n = C \tag{18}$$

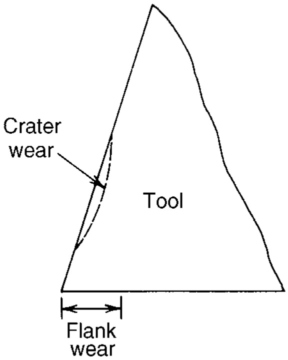


Fig. 5 Types of tool wear.

Table 4 Average Values of *n*

Tool Material	Work Material	n
HSS (18-4-1)	Steel	0.15
	C.I.	0.25
	Light metals	0.40
Cemented carbide	Steel	0.30
	C.I.	0.25
Sintered carbide	Steel	0.50
Ceramics	Steel	0.70

where V = cutting speed, fpm (m/sec)
T = tool life, min (sec)
n = exponent depending on cutting condition
C = constant, the cutting speed for a tool life of 1 min

Table 4 gives the approximate ranges for the exponent n. Taylor's equation is equivalent to

$$\log V = C - n \log T \tag{19}$$

which when plotted on log–log paper gives a straight line, as shown in Fig. 6.

Equation (20) incorporates the size of cut:

$$K = VT^n f^{n1} d^{n2} \tag{20}$$

Average values for n_1 = .5–.8
n_2 = .2–.4

Equation (21) incorporates the hardness of the workpiece:

$$K = VT^n f^{n1} d^{n2} (BHN)^{1.25} \tag{21}$$

4 METAL-CUTTING ECONOMICS

The efficiency of machine tools increases as cutting speeds increase, but tool life is reduced. The main objective of metal-cutting economics is to achieve the optimum conditions, that is, the minimum cost while considering the principal individual costs: machining cost, tool cost, tool-changing cost, and handling cost. Figure 7 shows the relationships among these four factors.

$$\text{machining cost} = C_o t_m \tag{22}$$

where C_o = operating cost per minute, which is equal to the machine operator's rate plus appropriate overhead
t_m = machine time in minutes, which is equal to $L/(fN)$, where L is the axial length of cut

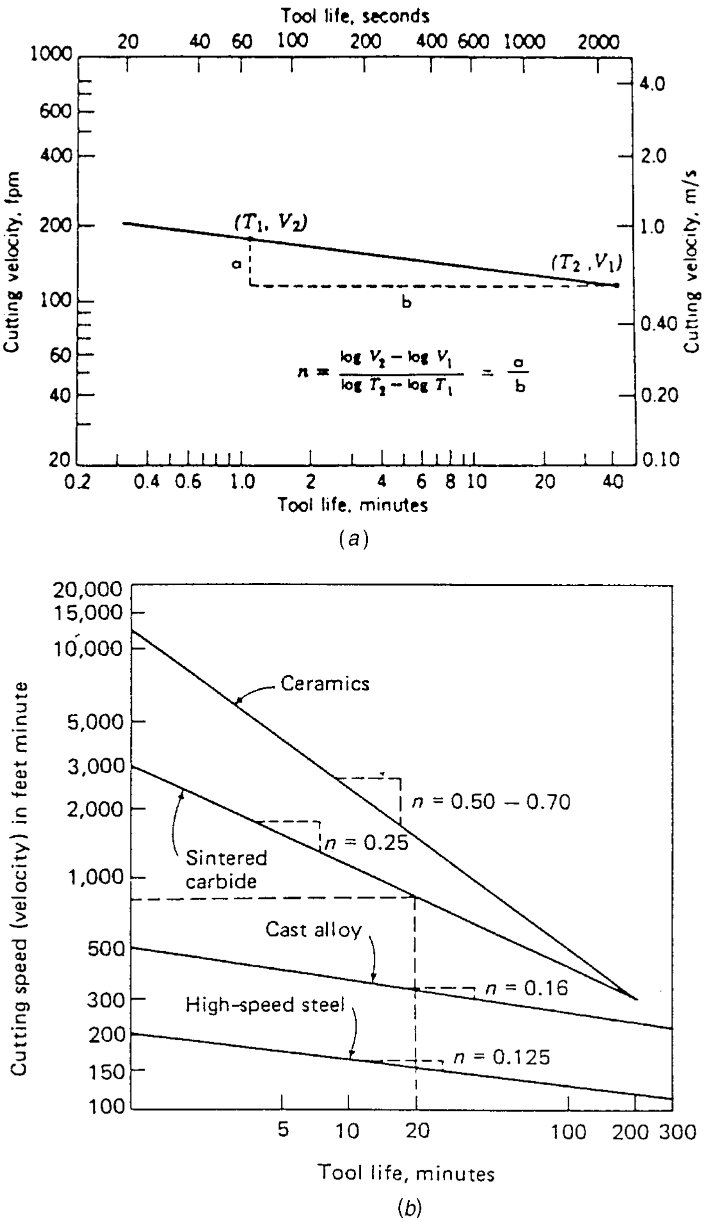
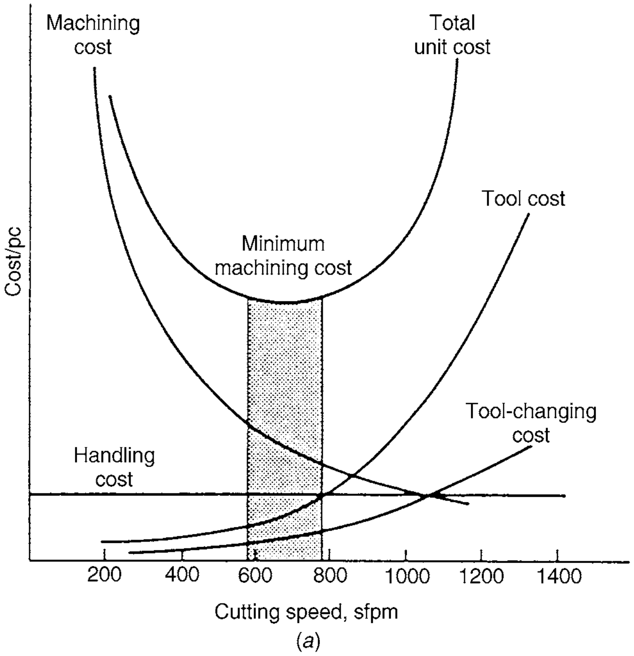


Fig. 6 Cutting speed/tool life relationship.

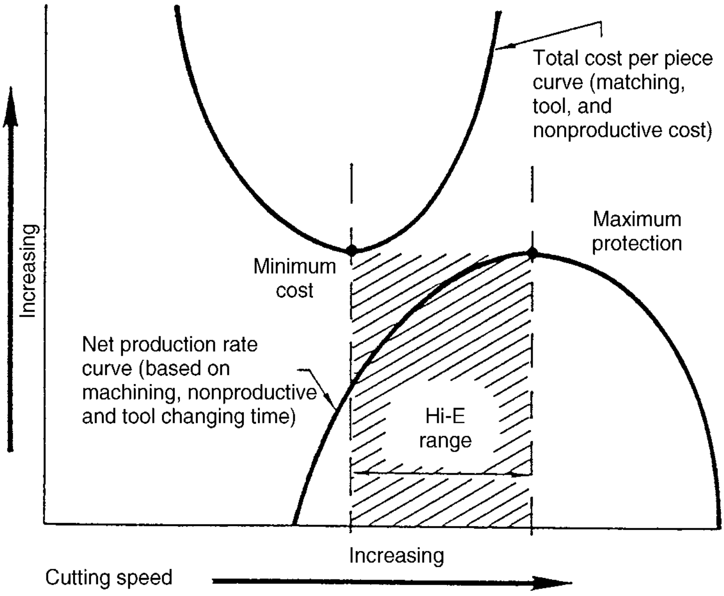
$$\text{tool cost per operation} = C_t \frac{t_m}{T} \tag{23}$$

where C_t = tool cost per cutting edge

T = tool life, which is equal to $(C/V)^{1/n}$



(*a*)



(*b*)

Fig. 7 Cost factors.

$$\text{tool changing cost} = C_o t_c (t_m / T) \tag{24}$$

where t_c = tool changing time, min

$$\text{handling cost} = C_o t_h$$

where t_h = handling time, min

The average unit cost C_u will be equal to

$$C_u = C_o t_m + \frac{t_m}{T}(C_t + C_o t_c) + C_o t_h \tag{25}$$

4.1 Cutting Speed for Minimum Cost ($V_{\min}$)

Differentiating the costs with respect to cutting speed and setting the results equal to zero will result in $V_{\min}$:

$$V_{\min} = \frac{C}{\left(\frac{1}{n} - 1\right)\left(\frac{C_o t + C_t}{C_o}\right)^n} \tag{26}$$

4.2 Tool Life Minimum Cost ($T_{\min}$)

Since the constant C is the same in Taylor's equation and Eq. (23), and if V corresponds to $V_{\min}$, then the tool life that corresponds to the cutting speed for minimum cost is

$$T_{\min} = \left(\frac{1}{n} - 1\right)\left(\frac{C_o t_c + C_t}{C_o}\right) \tag{27}$$

4.3 Cutting Speed for Maximum Production ($V_{\max}$)

This speed can be determined from Eq. (26) for the cutting speed for minimum cost by assuming that the tool cost is negligible, that is, by setting $C_1 = 0$:

$$V_{\max} = \frac{C}{\left[\left(\frac{1}{n} - 1\right) t_c\right]^n} \tag{28}$$

4.4 Tool Life for Maximum Production ($T_{\max}$)

By analogy to Taylor's equation, the tool life that corresponds to the maximum production rate is given by

$$T_{\max} = \left(\frac{1}{n} - 1\right) t_c \tag{29}$$

5 CUTTING-TOOL MATERIALS

The desirable properties for any tool material include the ability to resist softening at high temperature, which is known as red hardness; a low coefficient of friction; wear resistance; sufficient toughness and shock resistance to avoid fracture; and inertness with respect to workpiece material.

The principal materials used for cutting tools are carbon steels, cast nonferrous alloys, carbides, ceramic tools or oxides, and diamonds.

1. *High-carbon steels* contain (0.8–1.2%) carbon. These steels have good hardening ability, and with proper heat treatment hold a sharp cutting edge where excessive abrasion and high heat are absent. Because these tools lose hardness at around 600°F (315°C), they are not suitable for high speeds and heavy-duty work.
2. *High-speed steels* (HSS) are high in alloy contents such as tungsten, chromium, vanadium, molybdenum, and cobalt. High-speed steels have excellent hardenability and will retain a keen cutting edge to temperatures around 1200°F (650°C).
3. *Cast nonferrous alloys* contain principally chromium, cobalt, and tungsten, with smaller percentages of one or more carbide-forming elements, such as tantalum, molybdenum, or boron. Cast-alloy tools can maintain good cutting edges at temperatures up to 1700°F (935°C) and can be used at twice the cutting speed as HSS and still maintain the same feed. Cast alloys are not as tough as HSS and have less shock resistance.
4. *Carbides* are made by powder-metallurgy techniques. The metal powders used are tungsten carbide (WC), cobalt (Co), titanium carbide (TiC), and tantalum carbide (TaC) in different ratios. Carbide will maintain a keen cutting edge at temperatures over 2200°F (1210°C) and can be used at speeds two or three times those of cast alloy tools.
5. *Coated tools,* cutting tools, and inserts are coated by titanium nitride (TiN), titanium carbide (TiC), titanium carbonitride (TiCN), aluminum oxide (Al_2O_3), and diamond. Cutting speeds can be increased by 50% due to coating.
6. *Ceramic or oxide tool* inserts are made from aluminum oxide (Al_2O_3) grains with minor additions of titanium, magnesium, or chromium oxide by powder-metallurgy techniques. These inserts have an extremely high abrasion resistance and compressive strength, lack affinity for metals being cut, resistance to cratering and heat conductivity. They are harder than cemented carbides but lack impact toughness. The ceramic tool softening point is above 2000°F (1090°C) and these tools can be used at high speeds (1500–2000 ft/min) with large depth of cut. Ceramic tools have tremendous potential because they are composed of materials that are abundant in the earth's crust. Optimum cutting conditions can be achieved by applying negative rank angles (5–7°), rigid tool mountings, and rigid machine tools.
7. *Cubic boron nitride* (CBN) is the hardest material presently available, next to diamond. CBN is suitable for machining hardened ferrous and high-temperature alloys. Metal removal rates up to 20 times those of carbide cutting tools were achieved.
8. *Single-crystal diamonds* are used for light cuts at high speeds of 1000–5000 fpm to achieve good surface finish and dimensional accuracy.

They are used also for hard materials difficult to cut with other tool material.

9. *Polycrystalline diamond* cutting tools consist of fine diamond crystals, natural or synthetic, that are bonded together under high pressure and temperature. They are suitable for machining nonferrous metals and nonmetallic materials.

5.1 Cutting-Tool Geometry

The shape and position of the tool relative to the workpiece have a very important effect in metal cutting. There are six single-point tool angles critical to the machining process. These can be divided into three groups.

Rake angles affect the direction of chip flow, the characteristics of chip formation, and tool life. Positive rake angles reduce the cutting forces and direct the chip flow away from the material. Negative rake angles increase cutting forces but provide greater strength, as is recommended for hard materials.

Relief angles avoid excessive friction between the tool and workpiece and allow better access of coolant to tool–work interface.

The *side cutting-edge angle* allows the full load of the cut to be built up gradually. The *end cutting-edge angle* allows sufficient clearance so that the surface of the tool behind the cutting point will not rub over the work surface.

The purpose of the *nose radiuses* is to give a smooth surface finish and to increase the tool life by increasing the strength of the cutting edge. The elements of the single-point tool are written in the following order: back rake angle, side rake angle, end relief angle, side relief angle, end cutting-edge angle, side cutting-edge angle, and nose radius. Figure 8 shows the basic tool geometry.

Cutting tools used in various machining operations often appear to be very different from the single-point tool in Figure 8. Often they have several cutting edges, as in the case of drills, broaches, saws, and milling cutters. Simple analysis will show that such tools are comprised of a number of single-point cutting edges arranged so as to cut simultaneously or sequentially.

5.2 Cutting Fluids

The major roles of the cutting fluids—liquids or gases—are

1. Removal of the heat friction and deformation
2. Reduction of friction among chip, tool, and workpiece
3. Washing away chips
4. Reduction of possible corrosion on both workpiece and machine
5. Prevention of built-up edges

Cutting fluids work as coolants and lubricants. Cutting fluids applied depend primarily on the kind of material being used and the type of operation. The four major types of cutting fluids are

1. Soluble oil emulsions with water-to-oil ratios of 20:1 to 80:1
2. Oils
3. Chemicals and synthetics
4. Air

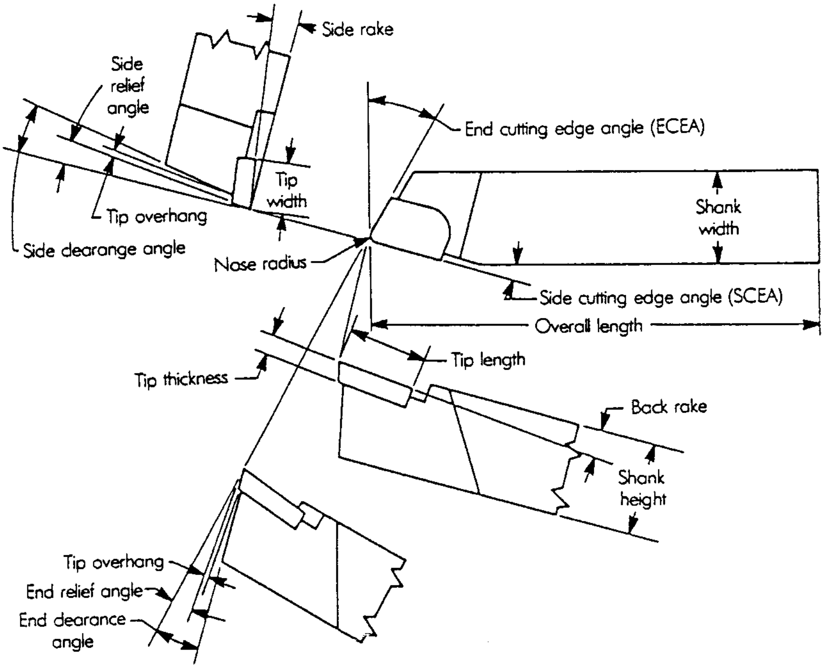


Fig. 8 Basic tool geometry.

At low cutting speeds (40 ft/min and below), oils are highly recommended, especially in tapping, reaming, and gear and thread machining. Cutting fluids with the maximum specific heat, such as soluble oil emulsions, are recommended at high speeds.

5.3 Machinability

Machinability refers to a system for rating materials on the basis of their relative ability to be machined easily, long tool life, low cutting forces, and acceptable surface finish. Additives such as lead, manganese sulfide, or sodium sulfide with percentages less than 3% can improve the machinability of steel and copper-based alloys, such as brass and bronze. In aluminum alloys, additions up to 1–3% of zinc and magnesium improve their machinability.

5.4 Cutting Speeds and Feeds

Cutting speed is expressed in feet per minute (m/sec) and is the relative surface speed between the cutting tool and the workpiece. It may be expressed by the simple formula $CS = \pi DN/12$ fpm in., where D is the diameter of the workpiece in inches in case of turning or the diameter of the cutting tool in case of drilling, reaming, boring, and milling, and N is the revolutions per minute. If D is given in millimeters, the cutting speed is $CS = \pi DN/60{,}000$ m/sec.

Feed refers to the rate at which a cutting tool advances along or into the surface of the workpiece. For machines in which either the workpiece or the tool turns, feed is expressed in inches per revolution (ipr) (mm/rev). For reciprocating tools or workpieces, feed is expressed in inches per stroke (ips) (mm/stroke).

The recommended cutting speeds, and depth of cut that resulted from extensive research, for different combinations of tools and materials under different cutting conditions can be found in many references, including Society of Manufacturing Engineers (SME) publications such as *Tool and Manufacturing Engineers Handbook*;[1] *Machining Data Handbook*;[2] Metcut Research Associates, Inc.; *Journal of Manufacturing Engineers*; *Manufacturing Engineering Transactions*; *American Society for Metals* (*ASM*) *Handbook*;[3] *American Machinist's Handbook*;[4] *Machinery's Handbook*;[5] American Society of Mechanical Engineering (ASME) publications; Society of Automotive Engineers (SAE) Publications; and *International Journal of Machine Tool Design and Research.*

6 TURNING MACHINES

Turning is a machining process for generating external surfaces of revolution by the action of a cutting tool on a rotating workpiece, usually held in a lathe. Figure 9 shows some of the external operations that can be done on a lathe. When the same action is applied to internal surfaces of revolution, the process is termed *boring*. Operations that can be performed on a lathe are turning, facing, drilling, reaming, boring, chamfering, taping, grinding, threading, tapping, and knurling.

The primary factors involved in turning are speed, feed, depth of cut, and tool geometry. Figure 10 shows the tool geometry along with the feed (f) and depth of cut (d). The cutting speed (CS) is the surface speed in feet per minute (sfm) or meters per sec (m/s). The feed (f) is expressed in inches of tool advance per revolution of the spindle (ipr) or (mm/rev). The depth of cut (d) is expressed in inches. Table 5 gives some of the recommended speeds while using HSS tools and carbides for the case of finishing and rough machining. The cutting speed (fpm) is calculated by

$$CS = \frac{\pi DN}{12} \text{ fpm} \tag{30}$$

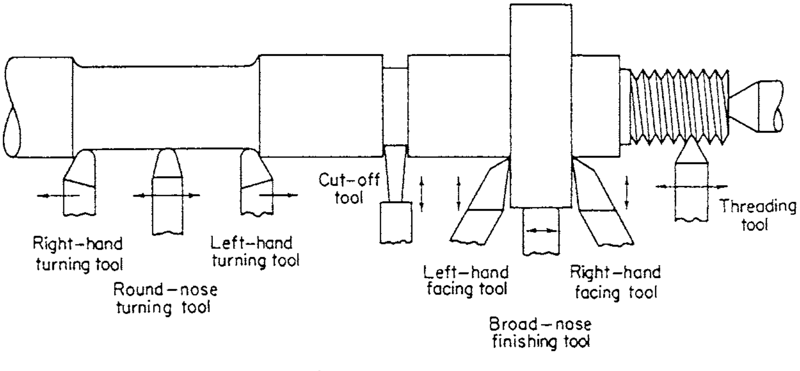


Fig. 9 Common lathe operations.

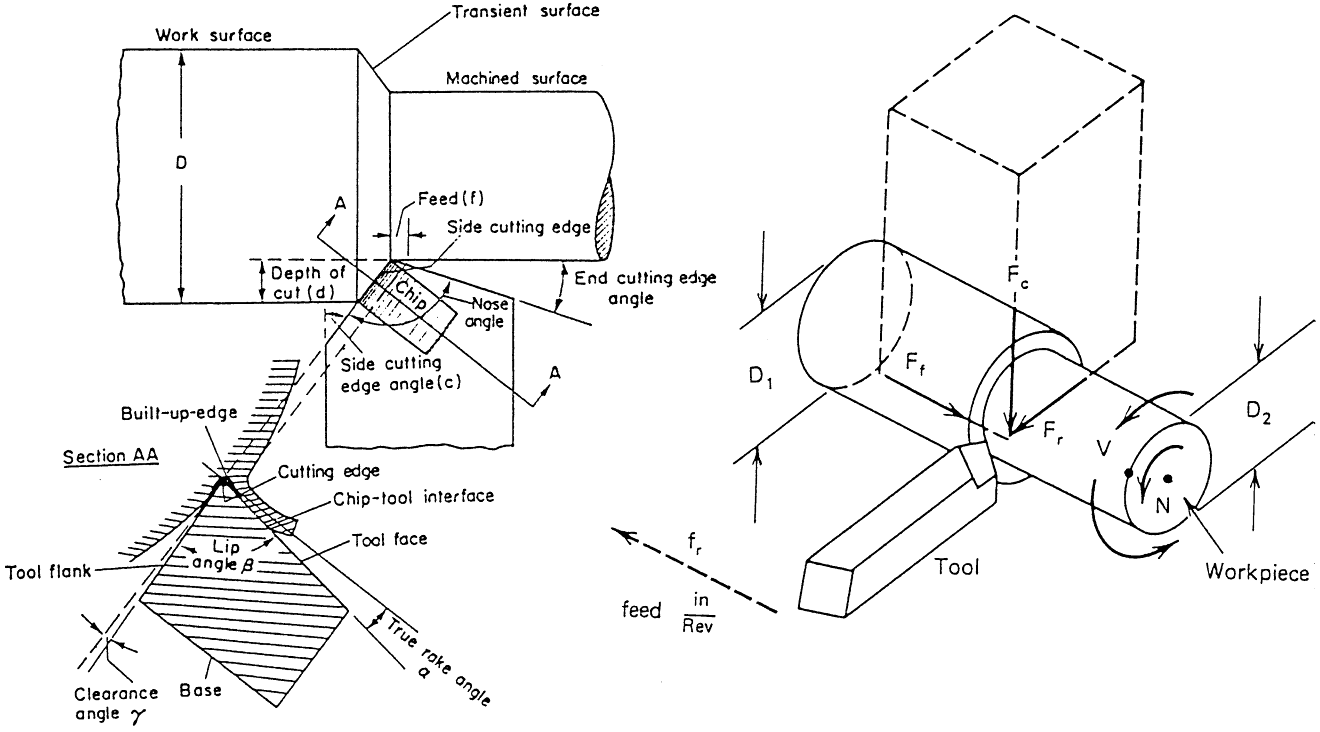


Fig. 10 Tool geometry—external turning.

Table 5 Typical Cutting Speeds ft/min (m/sec)

	High-Speed Steel		Carbide	
Material	Finish[a]	Rough[b]	Finish[a]	Rough[b]
Free cutting steels, 1112, 1315	250–350 (1.3–1.8)	80–160 (0.4–0.8)	600–750 (3.0–3.8)	350–500 (1.8–2.5)
Carbon steels, 1010, 1025	225–300 (1.1–1.5)	80–130 (0.4–0.6)	550–700 (2.8–3.5)	300–450 (1.5–2.3)
Medium steels, 1030, 1050	200–300 (1.0–1.5)	70–120 (0.4–0.6)	450–600 (2.3–3.0)	250–400 (1.3–2.0)
Nickel steels, 2330	200–300 (1.0–1.5)	70–110 (0.4–0.6)	425–550 (2.1–2.8)	225–350 (1.1–1.8)
Chromium nickel, 3120, 5140	150–200 (0.8–1.0)	60–80 (0.3–0.4)	325–425 (1.7–2.1)	175–300 (0.9–1.5)
Soft gray cast iron	120–150 (0.6–0.8)	80–100 (0.4–0.5)	350–450 (1.8–2.3)	200–300 (1.0–1.5)
Brass, normal	275–350 (1.4–1.8)	150–225 (0.8–1.1)	600–700 (3.0–3.5)	400–600 (2.0–3.0)
Aluminum	225–350 (1.1–1.8)	100–150 (0.5–0.8)	450–700 (2.3–3.5)	200–350 (1.0–1.8)
Plastics	300–500 (1.5–2.5)	100–200 (0.5–1.0)	400–650 (2.0–3.3)	150–300 (0.8–1.5)

[a] Cut depth, 0.015–0.10 in. (0.38–2.54 mm); feed 0.005–0.015 ipr (0.13–0.38 mm/rev).

[b] Cut depth, 0.20–0.40 in. (5.0–10.0 mm); feed, 0.030–0.060 ipr (0.75–1.5 mm/rev).

where D = workpiece diameter, in.
N = spindle revolutions per minute

For SI units,

$$CS = \frac{\pi DN}{1000} \quad \text{m/s} \tag{31}$$

where D is in mm
N is in revolutions per second

The tool advancing rate is $F = f \times N$ ipm (mm/sec). The machining time (T_1) required to turn a workpiece of length L in. (mm) is calculated from

$$T_1 = \frac{L}{F} \quad \text{min (sec)} \tag{32}$$

The machining time (T_2) required to face a workpiece of diameter D is given by

$$T_2 = \frac{D/2}{F} \quad \text{min (sec)} \tag{33}$$

The rate of metal removal (MRR) (Q) is given by

$$Q = 12fdCS \qquad \text{in.}^3/\text{min} \tag{34}$$

$$\text{Power} = QHP_{\mu} \qquad HP \tag{35}$$

$$\begin{aligned} \text{Power} &= \text{Torque } 2\pi N \\ &= \frac{\text{Torque} \times N}{63{,}025} \quad HP \end{aligned} \tag{36}$$

where torque is in in.–lbf

For SI units,

$$\text{Power} = \frac{\text{Torque} \times N}{9549} \quad KW \tag{37}$$

where torque is in newton–meter and N in rev/min
torque $= F_c \times R$

$$F_c = \frac{\text{Torque}}{R} \tag{38}$$

where R = radius of workpiece

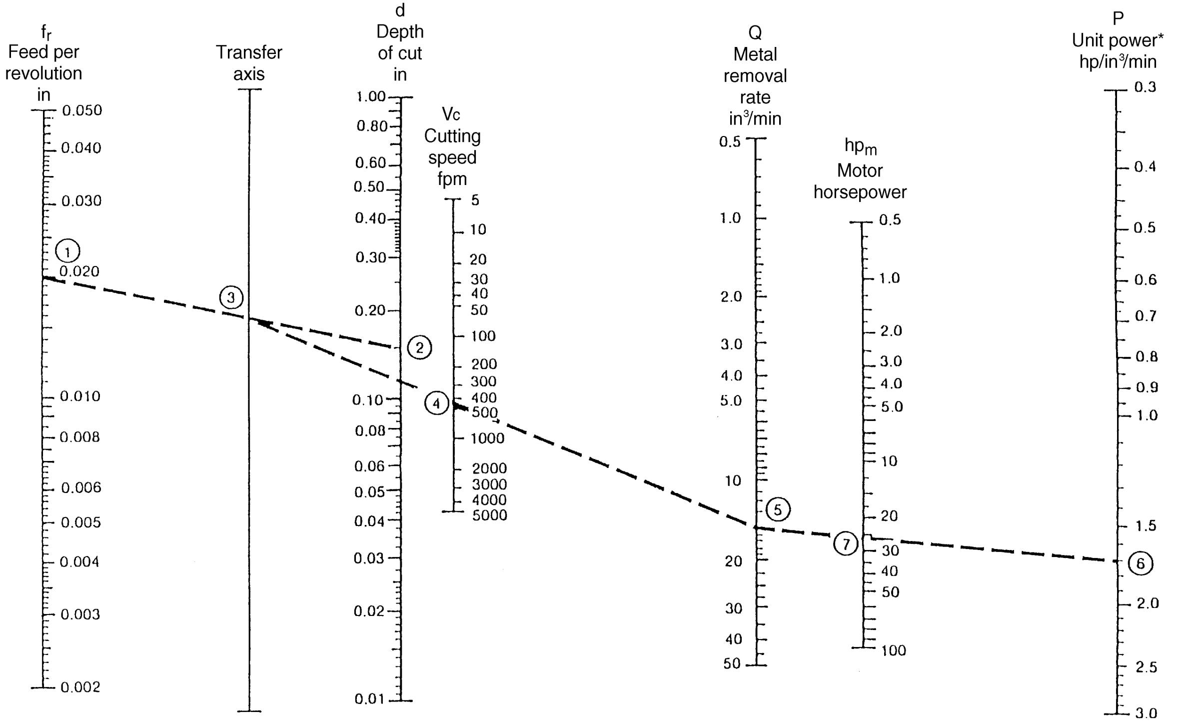
To convert to SI units,

$$\begin{aligned} \text{HP} \times 746 &= \text{? Watt (W)} \\ \text{f (lb)} \times 4.448 &= \text{? newtons} \\ \text{torque (in.–lb)} \times 0.11298 &= \text{? newton–meter (Nm)} \\ \text{HP/(cu in./min)} \times 2.73 &= \text{? W/(cu mm/sec)} \\ \text{ft/min} \times .00508 &= \text{? m/sec} \\ \text{in.}^3 \times 16{,}390 &= \text{? mm}^3 \end{aligned}$$

Alignment charts were developed for determining metal removal rate and motor power in turning. Figures 11 and 12 show the method of using these charts either for English or metric units. The unit power (P) is the adjusted unit power with respect to turning conditions and machine efficiency.

6.1 Lathe Size

The size of a lathe is specified in terms of the diameter of the work it will swing and the workpiece length it can accommodate. The main types of lathes are engine, turret, single-spindle automatic, automatic screw machine, multispindle automatic, multistation machines, boring, vertical, and tracer. The level of automation can range from semiautomatic to tape-controlled machining centers.



Horsepower = 12 x Feed x Depth X Speed X Unit Power

Example:

f_r = 0.020 in   d = 0.150 in

V_c = 425 fpm   P = 1.70 hp/in³/min

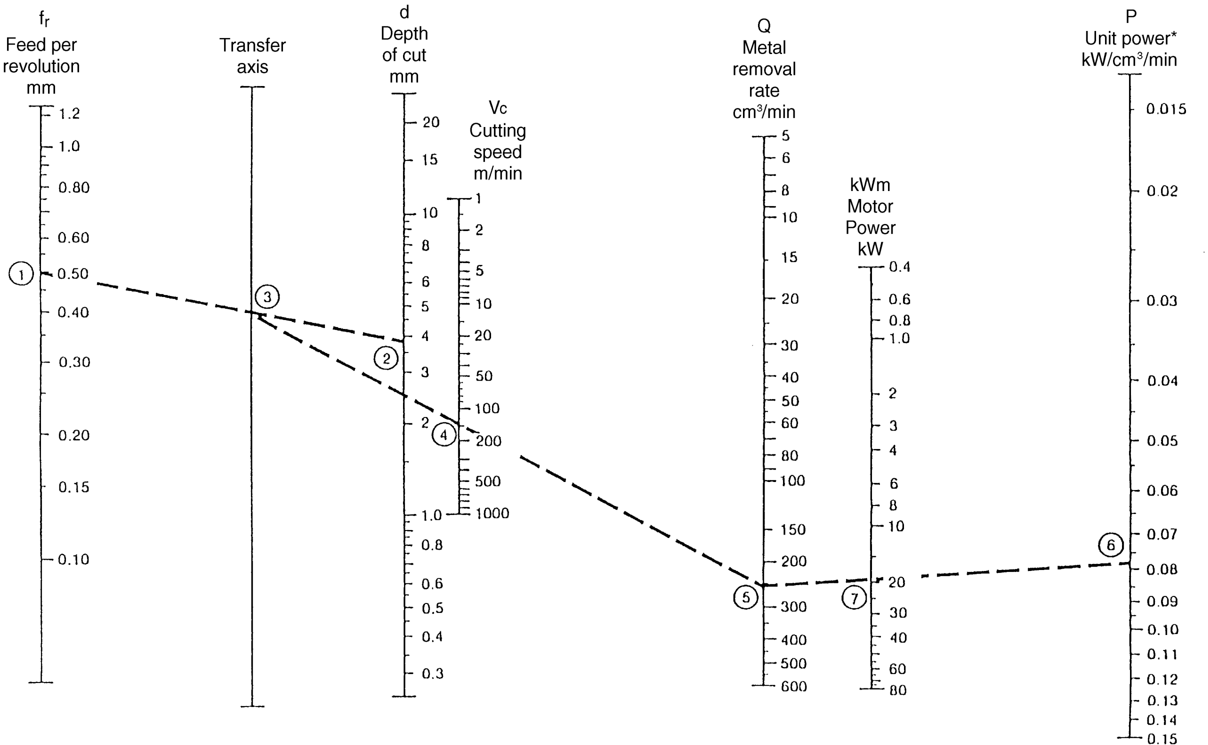
To Determine Motor Horsepower:

Connect Feed ① with Depth of cut ② to obtain point ③ on Transfer axis.

Connect ③ with Cutting speed ④ to obtain point ⑤ on Metal removal rate scale.

Connect point ⑤ with Unit power ⑥ to obtain 26 Motor horsepower at point ⑦.

Fig. 11 Alignment chart for determining metal removal rate and motor horsepower in turning—English units.



Power = Feed x Depth x Speed x Unit power

Example:

f_r = 0.5 mm d = 3.8 mm

V_c = 130 m/min P = 0.077 kW/cm³/min

To Determine Motor Horsepower:

Connect Feed ① with Depth of cut ② to obtain point ③ on Transfer axis.

Connect ③ with Cutting speed ④ to obtain point ⑤ on Metal removal rate scale.

Connect point ⑤ with Unit power ⑥ to obtain 19.02 kW at Motor, point ⑦.

Fig. 12 Alignment chart for determining metal removal rate and motor power in turning—metric units.

6.2 Break-Even (BE) Conditions

The selection of a specific machine for the production of a required quantity q must be done in a way to achieve minimum cost per unit produced. The incremental setup cost is given by ΔC_t, C_1 is the machining cost per unit on the first machine, and C_2 is the machining cost for the second machine, the break-even point will be calculated as follows:

$$BE = \Delta C \sqrt{(C_1 - C_2)}$$

7 DRILLING MACHINES

Drills are used as the basic method of producing holes in a wide variety of materials. Figure 13 indicates the nomenclature of a standard twist drill and its comparison with a single-point tool. Knowledge of the thrust force and torque developed in the drilling process is important for design consideration. Figure 14 shows the forces developed during the drilling process. From the force diagram, the thrust force must be greater than $2P_y + P_y^1$ to include the friction on the sides and to be able to penetrate in the metal. The torque required is equal to P_2X. It is reported in the *Tool and Manufacturing Engineers Handbook*[1] that the following relations reasonably estimate the torque and thrust requirements of sharp twist drills of various sizes and designs.

Torque:

$$M = KF^{0.8}d^{1.8}A \quad \text{in.–lbf} \tag{39}$$

Thrust:

$$T = 2Kf^{0.8}d^{0.8}B + kd^2E \quad \text{lb} \tag{40}$$

The thrust force has a large effect upon the required strength, rigidity, and accuracy, but the power required to feed the tool axially is very small.

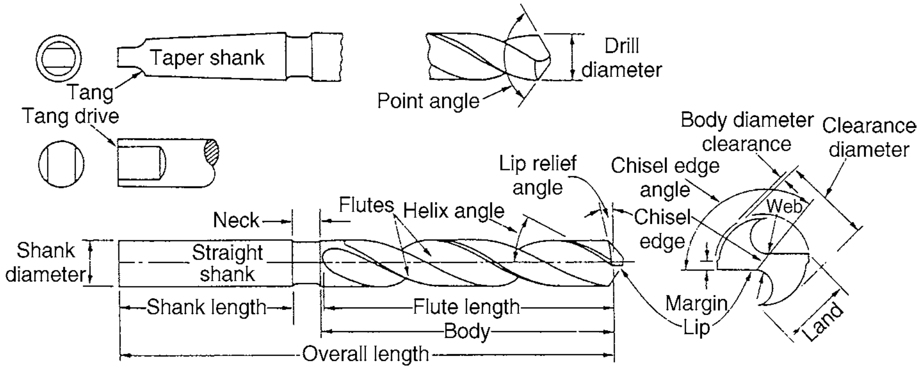


Fig. 13 Drill geometry.

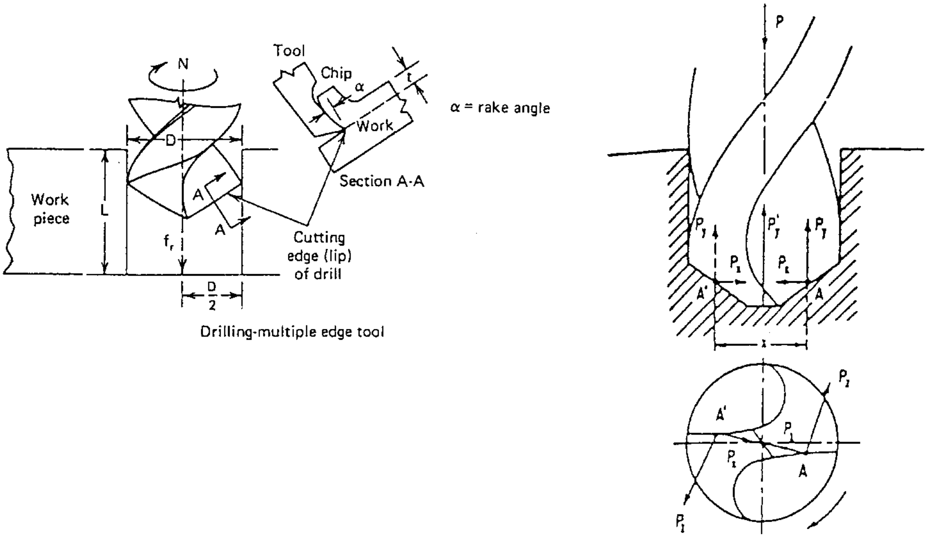


Fig. 14 Thrust forces and torque in drilling operation.

Cutting power:

$$HP = \frac{MN}{63{,}025} \tag{41}$$

where K = work–material constant
f = drill feed, ipr
d = drill diameter, in.
A, B, E = design constants
N = drill speed, rpm

Tables 6 and 7 give the constants used with the previous equations. Cutting speed at the surface is usually taken as 80% of turning speeds and is given by

Table 6 Work-Material Constants for Calculating Torque and Thrust (National Twist Drill)

Work Material	K
Steel, 200 Bhn	24,000
Steel, 300 Bhn	31,000
Steel, 400 Bhn	34,000
Most aluminum alloys	7,000
Most magnesium alloys	4,000
Most brasses	14,000
Leaded brass	7,000
Cast iron, 65 Bhn	15,000
Free-machining mild steel, resulfurized	18,000
Austenitic stainless steel (type 316)	34,000

Table 7 Torque and Thrust Constants Based on Ratios *c/d* or *w/d* (National Twist Drill)

c/d	*w/d*	Torque Constant A	Thrust Constant B	Thrust Constant E
0.03	0.025	1.000	1.100	0.001
0.05	0.045	1.005	1.140	0.003
0.08	0.070	1.015	1.200	0.006
0.10	0.085	1.020	1.235	0.010
0.13	0.110	1.040	1.270	0.017
0.15	0.130	1.080	1.310	0.022
0.18	0.155	1.085	1.355	0.030
0.20	0.175	1.105	1.380	0.040
0.25	0.220	1.155	1.445	0.065
0.30	0.260	1.235	1.500	0.090
0.35	0.300	1.310	1.575	0.120
0.40	0.350	1.395	1.620	0.160

$$CS = \frac{\pi d N}{12} \quad \text{fpm}$$

Force in cutting direction:

$$F_c = \frac{33{,}000\ HP}{CS} \quad \text{lb} \tag{42}$$

For SI units,

$$CS = \frac{\pi d_1 N}{60{,}000} \quad \text{m/sec} \tag{43}$$

c = chisel-edge length, in.

d = drill diameter, in.

w = web thickness, in.

d_1 = drill diameter, in mm

Unit HP (hp/in.^{3}/min) × 2.73 = ? unit power (kW/cm^3/s)

$$\text{kW} = \frac{MN}{9549} \tag{44}$$

$$M = \text{torque} \quad \text{Nm}$$

For drills of regular proportion the ratio c/d is $= 0.18$ and $c = 1.15\ w$, approximately.

It is a common practice to feed drills at a rate that is proportional to the drill diameter in accordance with

$$f = \frac{d}{65} \tag{45}$$

For holes that are longer than 3d, feed should be reduced. Also feeds and speeds should be adjusted due to differences in relative chip volume, material structure, cutting fluid effectiveness, depth of hole, and conditions of drill and machine. The advancing rate is

$$F = f \times N \quad \text{ipm} \tag{46}$$

The recommended feeds are given in Table 8.

The time T required to drill a hole of depth h is given by

$$T = \frac{h + 0.3d}{F} \quad \text{min} \tag{47}$$

The extra distance of $0.3d$ is approximately equal to the distance from the tip to the effective diameter of the tool. The rate of metal removal in case of blind holes is given by

$$Q = \left(\frac{\pi d^2}{4}\right) F \quad \text{in.}^3/\text{min} \tag{48}$$

When torque is unknown, the horsepower requirement can be calculated by

$$HP_c = Q \times C \times W \times (HP_\mu) \quad \text{hp}$$

C, W, HP_μ are given in previous sections.

$$\text{Power} = HP_c \times 396{,}000 \quad \text{in.–lb/min} \tag{49}$$

$$\text{Torque} = \frac{\text{Power}}{2\pi N} \quad \text{in–lbf}$$

$$F_c = \frac{\text{Torque}}{R} \quad \text{lb}$$

Along the cutting edge of the drill, the cutting speed is reduced toward the center

Table 8 Recommended Feeds for Drills

Diameter		Feed	
(in.)	(mm)	(ipr)	(mm/rev)
Under ⅛	3.2	0.001–0.002	0.03–0.05
⅓–¼	3.2–6.4	0.002–0.004	0.05–0.10
¼–½	6.4–12.7	0.004–0.007	0.10–0.18
½–1	12.7–25.4	0.007–0.015	0.18–0.38
Over 1	25.4	0.015–0.025	0.38–0.64

as the diameter is reduced. The cutting speed is actually zero at the center. To avoid the region of very low speed and to reduce high thrust forces that might affect the alignment of the finished hole, a pilot hole is usually drilled before drilling holes of medium and large sizes. For the case of drilling with a pilot hole

$$\begin{aligned} Q &= \frac{\pi}{4}(d^2 - d_p^2)F \\ &= \frac{\pi}{4}(d + d_p)(d - d_p)F \quad \text{in.}^3/\text{min} \end{aligned} \tag{50}$$

Due to the elimination of the effects of the chisel-edge region, the equations for torque and thrust can be estimated as follows:

$$M_p = M\left[\frac{1 - \left(\dfrac{d_p}{d}\right)^2}{\left(1 + \dfrac{d_1}{d}\right)^{0.2}}\right] \tag{51}$$

$$T_p = T\left[\frac{1 - \dfrac{d_1}{d}}{\left(1 + \dfrac{d_1}{d}\right)^{0.2}}\right] \tag{52}$$

where d_p = pilot hole diameter

Alignment charts were developed for determining motor power in drilling. Figures 15 and 16 show the use of these charts either for English or metric units. The unit power* (P) is the adjusted unit power with respect to drilling conditions and machine efficiency.

For English units,

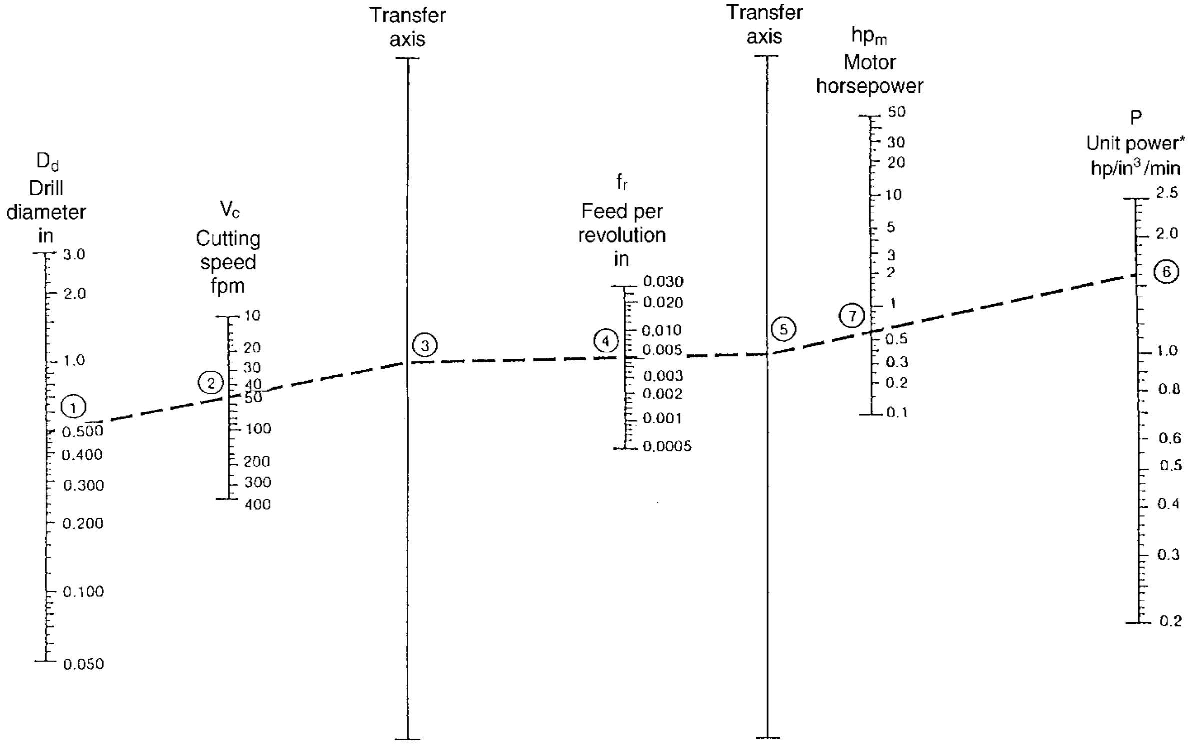
$$HP_m = \frac{\pi D^2}{4} \times f \times N \times P^*$$

$$N = \frac{12V}{\pi D}$$

As

$$\begin{aligned} HP_m &= \frac{\pi D^2}{4} \times f \times \frac{12V}{\pi D} \times P^* \\ &= 3\,D \times f \times V \times P^* \end{aligned}$$

For metric units,



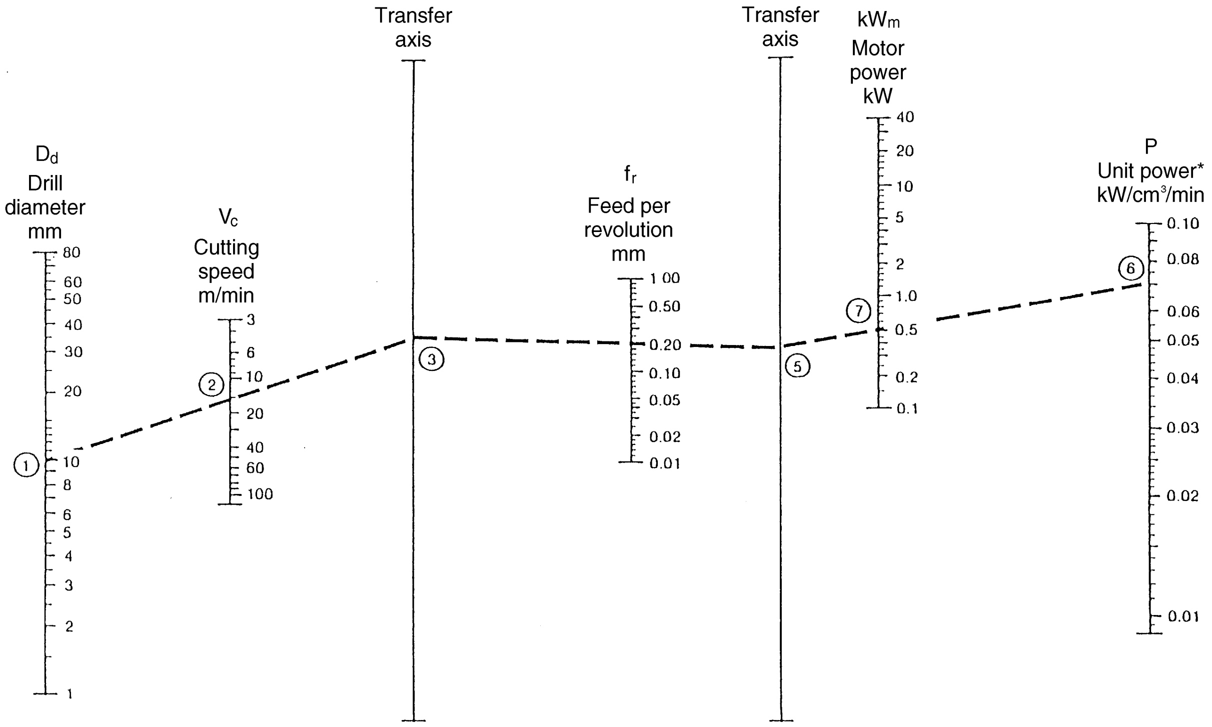
Horsepower = 3 x Drill diameter x Speed x Feed x Unit power

Example:
D_d = 0.500 in
V_c = 50 fpm
f_r = 0.005 in
P = 1.6 hp/in^3/min

To Determine Motor Horsepower:

Connect Drill diameter ① with Cutting speed ② to obtain point ③ on Transfer axis. Connect ③ with Feed ④ to obtain point ⑤ on Transfer axis. Connect ⑤ with Unit power ⑥ to obtain 0.60 Motor horsepower, point ⑦.

Fig. 15 Alignment chart for determining motor horsepower in drilling—English units.



Motor Power = 0.25 x Drill diameter x Speed x Feed x Unit Power

Example:

D_d = 10 mm

V_c = 15 m/min

f_r = 0.2 mm

P = 0.07 kW/cm³/min

To Determine Motor Horsepower:

Connect Drill diameter ① with Cutting speed ② to obtain point ③ on Transfer axis. Connect ③ with Feed ④ to obtain point ⑤ on Transfer axis. Connect ⑤ with Unit power ⑥ to obtain 0.53 kW at motor power, point ⑦.

Fig. 16 Alignment chart for determining motor power in drilling—metric units.

$$HP_m = \frac{\pi D^2}{4 \times 100} \times \frac{f}{10} \times N \times P$$

$$P^* \text{ in kW/cm}^3\text{/min}$$

$$N = \frac{1000V}{\pi D}$$

$$HP_m = \frac{\pi D^2}{4 \times 100} \times \frac{f}{10} \times \frac{100V}{\pi D} \times P^*$$

$$= 0.25\, D \times f \times V \times P^*$$

7.1 Accuracy of Drills

The accuracy of holes drilled with a two-fluted twist drill is influenced by many factors, including the accuracy of the drill point; the size of the drill, the chisel edge, and the jigs used; the workpiece material; the cutting fluid used; the rigidity and accuracy of the machine used; and the cutting speed. Usually, when drilling most materials, the diameter of the drilled holes will be oversize. Table 9 provided the results of tests reported by The Metal Cutting Tool Institute for holes drilled in steel and cast iron.

Gun drills differ from conventional drills in that they are usually made with a single flute. A hole provides a passageway for pressurized coolant, which serves as a means of both keeping the cutting edge cool and flushing out the chips, especially in deep cuts.

Spade drills (Fig. 17) are made by inserting a spade-shaped blade into a shank. Some advantages of spade drills are (1) efficiency in making holes up to 15 in. in diameter; (2) low cost, since only the insert is replaced; (3) deep hole drilling; and (4) easiness of chip breaking on removal.

Trepanning is a machining process for producing a circular hole, groove, disk, cylinder, or tube from solid stock. The process is accomplished by a tool containing one or more cutters, usually single-point, revolving around a center. The advantages of trepanning are (1) the central core left is solid material, not chips, which can be used in later work; and (2) the power required to produce a given hole diameter is highly reduced because only the annulus is actually cut.

Reaming, boring, counterboring, centering and countersinking, spotfacing, tapping, and chamfering processes can be done on drills. Microdrilling and submicrodrilling achieve holes in the range of 0.000025–0.20 in. in diameter.

Table 9 Oversize Diameters in Drilling

	Amount Oversize (in.)		
Drill Diameter (in.)	Average Max.	Mean	Average Min.
1⁄16	0.002	0.0015	0.001
1⁄8	0.0045	0.003	0.001
1⁄4	0.0065	0.004	0.0025
1⁄2	0.008	0.005	0.003
3⁄4	0.008	0.005	0.003
1	0.009	0.007	0.004

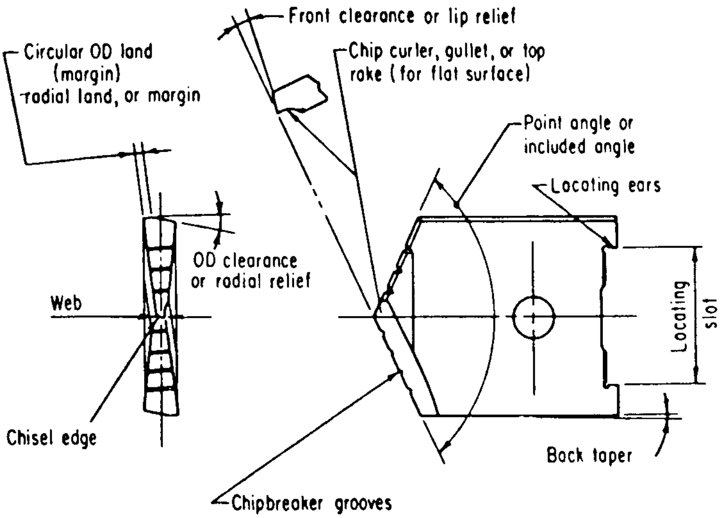


Fig. 17 Spade-drill blade elements.

Drilling machines are usually classified in the following manner:

1. Bench: plain or sensitive
2. Upright: single-spindle or turret
3. Radial
4. Gang
5. Multispindle
6. Deep-hole: vertical or horizontal
7. Transfer

8 MILLING PROCESSES

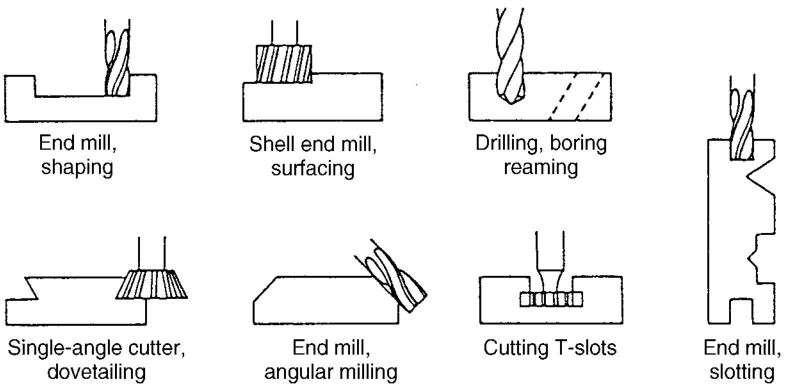
The milling machines use a rotary multitooth cutter that can be designed to mill flat or irregularly shaped surfaces, cut gears, generate helical shapes, drill, bore, or do slotting work. Milling machines are classified broadly as vertical or horizontal. Figure 18 shows some of the operations that are done on both types.

Feed in milling (F) is specified in inches per minute, but it is determined from the amount each tooth can remove or feed per tooth (f_t). The feed in./min is calculated from

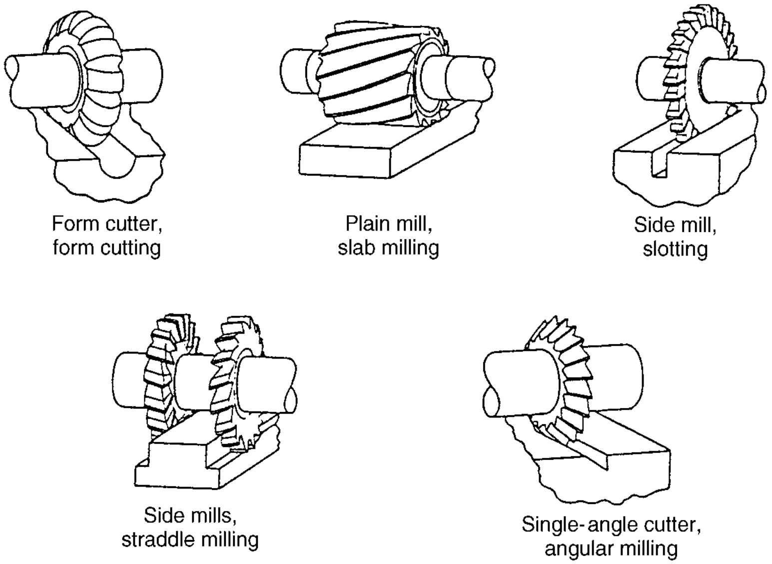
$$F = f_t \times n \times N \quad \text{in./min} \tag{53}$$

where n = number of teeth in cutter
N = rpm

Table 10 gives the recommended f_t for carbides and HSS tools. The cutting speed CS is calculated as follows:



(*a*) Vertical-milling-machine cuts



(*b*) Horizontal-milling-machine cuts

Fig. 18 Applications of (*a*) vertical; (*b*) horizontal milling machines.

Table 10 Recommended Feed per Tooth for Milling Steel with Carbide and HSS Cutters

	Feed per Tooth	
Type of Milling	Carbides	HSS
Face	0.008–0.015	0.010
Side or straddle	0.008–0.012	0.006
Slab	0.008–0.012	0008
Slotting	0.006–0.010	0.006
Slitting saw	0.003–0.006	0.003

Table 11 Table of Cutting Speeds (sfpm)–Milling

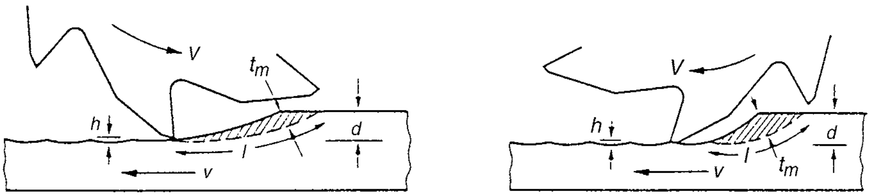
	HSS Tools		Carbide-Tipped Tools	
Work Material	Rough Mill	Finish Mill	Rough Mill	Finish Mill
Cast iron	50–60	80–110	180–200	350–400
Semisteel	40–50	65–90	140–160	250–300
Malleable iron	80–100	110–130	250–300	400–500
Cast steel	45–60	70–90	150–180	200–250
Copper	100–150	150–200	600	1000
Brass	200–300	200–300	600–1000	600–1000
Bronze	100–150	150–180	600	1000
Aluminum	400	700	800	1000
Magnesium	600–800	1000–1500	1000–1500	1000–5000
SAE steels				
1020 (coarse feed)	60–80	60–80	300	300
1020 (fine feed)	100–120	100–120	450	450
1035	75–90	90–120	250	250
X-1315	175–200	175–200	400–500	400–500
1050	60–80	100	200	200
2315	90–110	90–110	300	300
3150	50–60	70–90	200	200
4150	40–50	70–90	200	200
4340	40–50	60–70	200	200
Stainless steel	60–80	100–120	240–300	240–300
Titanium	30–70		200–350	

$$CS = \frac{\pi D N}{12} \text{ fpm}$$

where D = tool diameter, in.

Table 11 gives the recommended cutting speeds while using HSS and carbide-tipped tools. The relationship between cutter rotation and feed direction is shown in Fig. 19. In climb milling or down milling, the chips are cut to maximum thickness at initial engagement and decrease to zero thickness at the end of engagement. In conventional or up milling, the reverse occurs. Because of the initial impact, climb milling requires rigid machines with backlash eliminators.

The material removal rate (MRR) is $Q = F \times w \times d$, where w is the width of cut and d is the depth of cut. The horsepower required for milling is given by



(a) (b)

Fig. 19 Cutting action in up-and-down milling.

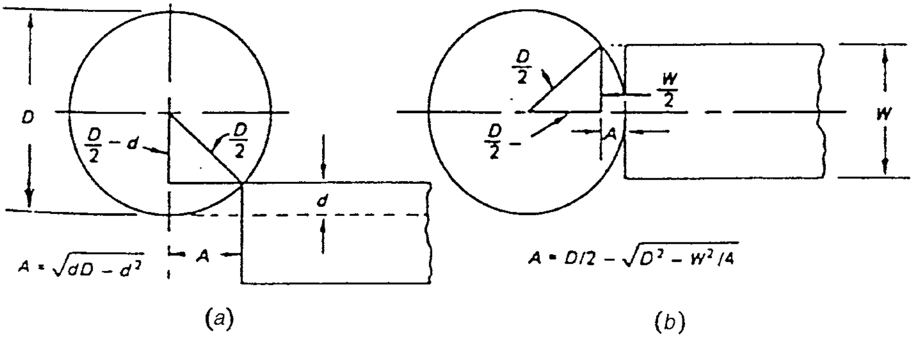


Fig. 20 Allowance for approach in (*a*) plain or slot milling; (*b*) face milling.

$$HP_c = HP_\mu \times Q$$

Machine horsepower is determined by

$$HP_m = \frac{HP_c}{Eff.} + HP_i \qquad (54)$$

where Hp_i = idle horsepower

Alignment charts were developed for determining metal removal rate (MRR) and motor power in face milling. Figures 21 and 22 show the method of using these charts either for English or metric units.

The time required for milling is equal to distance required to be traveled by the cutter to complete the cut (L_1) divided by the feed rate F. L_1 is equal to the length of cut (L) plus cutter approach A and the overtravel OT. The machining time T is calculated from

$$T = \frac{L + A + OT}{F} \text{ min} \qquad (55)$$

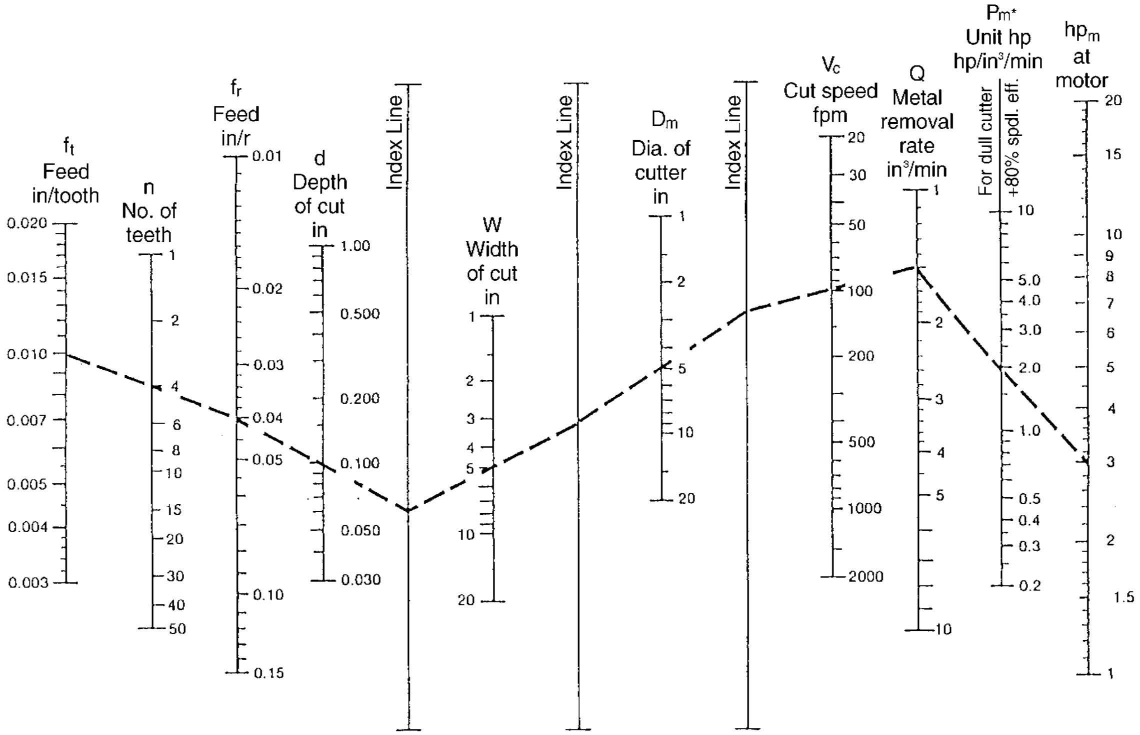
OT depends on the specific milling operation.

The milling machines are designed according to the longitudinal table travel. Milling machines are built in different types, including:

1. Column-and-knee: vertical, horizontal, universal, and ram
2. Bed-type, multispindle
3. Planer
4. Special, turret, profilers, and duplicators
5. Numerically controlled

9 GEAR MANUFACTURING

Gears are made by various methods, such as machining, rolling, extrusion, blanking, powder metallurgy, casting, or forging. Machining still is the unsurpassed



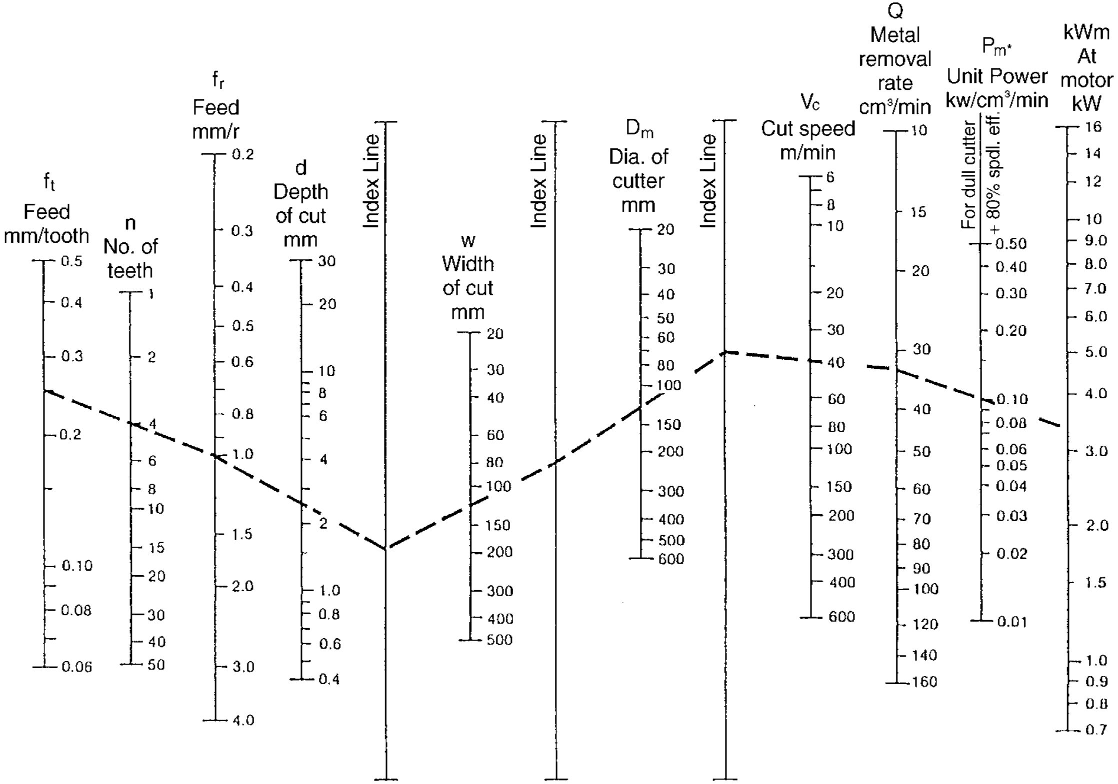
Example:
f_t = 0.010 in/tooth
n = 4 teeth
f_r = 0.04 in/r

d = 0.100 in
w = 5 in
D_m = 5 in

V_c = 100 fpm
Q = 1.53 in³/min
P = 2.0 hp/in³/min
hp_m = 3.0 hp

$$hp_m = \frac{P_m \times 3.82 \times f_t \times n \times d \times w \times V_c}{D_m}$$

Fig. 21 Alignment chart for determining metal removal rate and motor horsepower in face milling—English units.



Example:
f_t = 0.25 mm/tooth
n = 4 teeth
f_r = 1.0 mm/r
d = 2.5 mm
w = 125 mm
D_m = 125 mm
V_c = 40 m/min
Q = 31.8 cm³/min
P = 0.1 kW/cm³/min
kW_m = 3.18 kW

$$hp_m = \frac{P_m \times 318.3 \times f_t \times n \times d \times w \times V_c}{1000\, D_m}$$

Fig. 22 Alignment chart for determining metal removal rate and motor power in face milling—metric units.

method of producing gears of all types and sizes with high accuracy. Roll forming can be used only on ductile materials; however, it has been highly developed and widely adopted in recent years. Casting, powder metallurgy, extruding, rolling, grinding, molding, and stamping techniques are used commercially in gear production.

9.1 Machining Methods

There are three basic methods for machining gears.

Form cutting uses the principle illustrated in Fig. 23. The equipment and cutters required are relatively simple, and standard machines, usually milling,

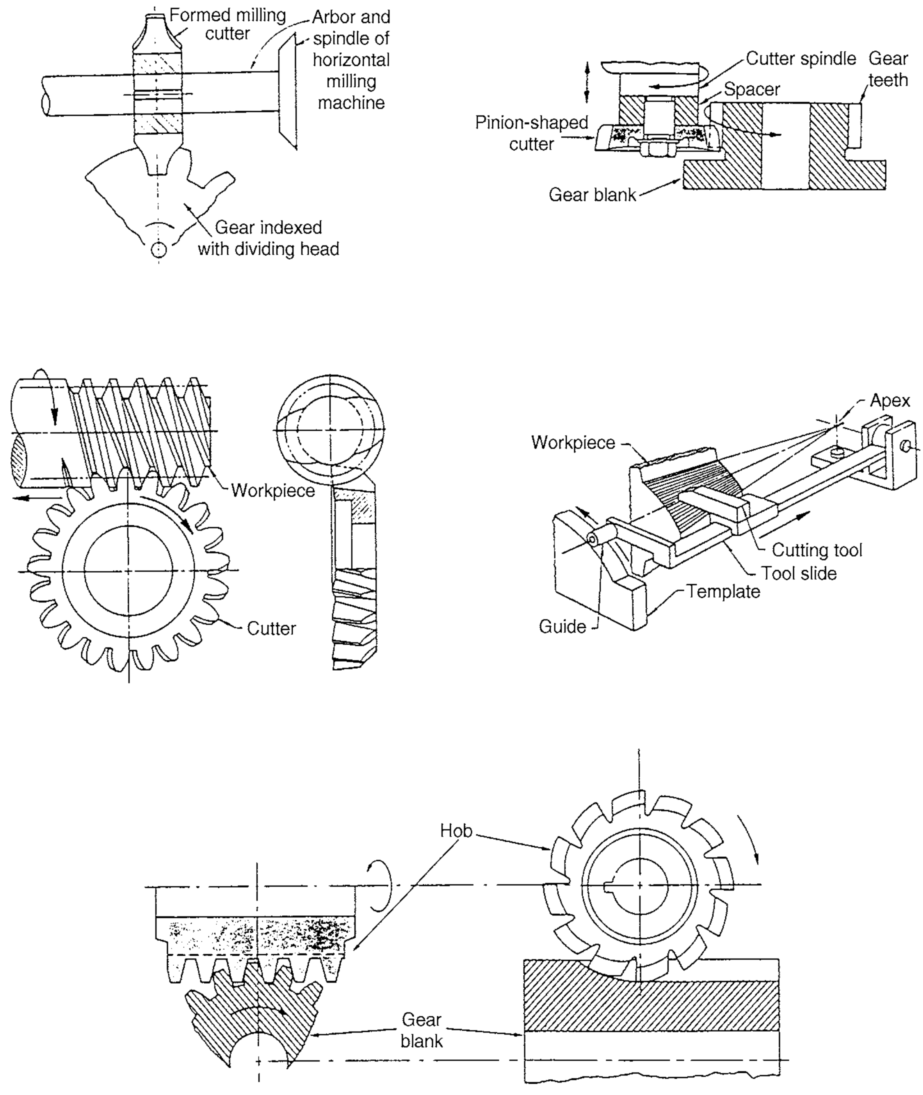


Fig. 23 Machining gears.

Table 12 Standard Gear Cutters

Cutter Number	Gear Tooth Range
1	135 teeth to rack
2	55–34
3	35–54
4	26–34
5	21–25
6	17–20
7	14–16
8	12–13

are often used. Theoretically, there should be different-shaped cutters for each size of gear for a given pitch, as there is a slight change in the curvature of the involute. However, one cutter can be used for several gears having different numbers of teeth without much sacrifice in their operating action. The eight standard involute cutters are listed in Table 12. On the milling machine, the index or dividing head is used to rotate the gear blank through a certain number of degrees after each cut. The rule to use is: turns of index handle $= 40/N$, where N is the number of teeth. Form cutting is usually slow.

Template machining utilizes a simple, single-point cutting tool that is guided by a template. However, the equipment is specialized, and the method is seldom used except for making large-bevel gears.

The *generating process* is used to produce most high-quality gears. This process is based on the principle that any two involute gears, or any gear and a rack, of the same diametral pitch will mesh together. Applying this principle, one of the gears (or the rack) is made into a cutter by proper sharpening and is used to cut into a mating gear blank and thus generate teeth on the blank. Gear shapers (pinion or rack), gear-hobbing machines, and bevel-gear generating machines are good examples of the gear generating machines.

9.2 Gear Finishing

To operate efficiently and have satisfactory life, gears must have accurate tooth profile and smooth and hard faces. Gears are usually produced from relatively soft blanks and are subsequently heat-treated to obtain greater hardness, if it is required. Such heat treatment usually results in some slight distortion and surface roughness. *Grinding and lapping* are used to obtain very accurate teeth on hardened gears. Gear-shaving and burnishing methods are used in gear finishing. Burnishing is limited to unhardened gears.

10 THREAD CUTTING AND FORMING

Three basic methods are used for the manufacturing of threads; *cutting, rolling,* and *casting.* Die casting and molding of plastics are good examples of casting. The largest number of threads are made by rolling, even though it is restricted to standardized and simple parts, and ductile materials. Large numbers of threads are cut by the following methods:

1. Turning
2. Dies: manual or automatic (external)

3. Milling
4. Grinding (external)
5. Threading machines (external)
6. Taps (internal)

10.1 Internal Threads

In most cases, the hole that must be made before an internal thread is tapped is produced by drilling. The hole size determines the depth of the thread, the forces required for tapping, and the tap life. In most applications, a drill size is selected that will result in a thread having about 75% of full thread depth. This practice makes tapping much easier, increases the tap's life, and only slightly reduces the resulting strength. Table 13 gives the drill sizes used to produce 75% thread depth for several sizes of UNC threads. The feed of a tap depends on the lead of the screw and is equal to 1/lead ipr.

Cutting speeds depend on many factors, such as

1. Material hardness
2. Depth of cut
3. Thread profile
4. Tooth depth
5. Hole depth
6. Fineness of pitch
7. Cutting fluid

Cutting speeds can range from lead 3 ft/min (1 m/min) for high-strength steels to 150 ft/min (45 m/min) for aluminum alloys. Long-lead screws with different configurations can be cut successfully on milling machines, as in Fig. 24. The feed per tooth is given by the following equation:

$$f_t = \frac{\pi d S}{n N} \tag{56}$$

Table 13 Recommended Tap-Drill Sizes for Standard Screw-Thread Pitches (American National Coarse-Thread Series)

Number or Diameter	Threads per Inch	Outside Diameter of Screw	Tap Drill Sizes	Decimal Equivalent of Drill
6	32	0.138	36	0.1065
8	32	0.164	29	0.1360
10	24	0.190	25	0.1495
12	24	0.216	16	0.1770
¼	20	0.250	7	0.2010
⅜	16	0.375	5⁄16	0.3125
½	13	0.500	27⁄64	0.4219
¾	10	0.750	21⁄32	0.6562
1	8	1.000	⅞	0.875

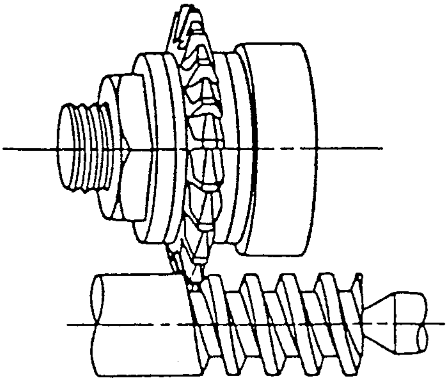


Fig. 24 Single-thread milling cutter.

where d = diameter of thread
n = number of teeth in cutter
N = rpm of cutter
S = rpm of work

10.2 Thread Rolling

In thread rolling, the metal on the cylindrical blank is cold-forged under considerable pressure by either rotating cylindrical dies or reciprocating flat dies. The advantages of thread rolling include improved strength, smooth surface finish, less material used (~19%), and high production rate. The limitations are that blank tolerance must be close, it is economical only for large quantities, it is limited to external threads, and it is applicable only for ductile materials, less than Rockwell C37.

11 BROACHING

Broaching is unique in that it is the only one of the basic machining processes in which the feed of the cutting edges is built into the tool. The machined surface is always the inverse of the profile of the broach. The process is usually completed in a single, linear stroke. A broach is composed of a series of single-point cutting edges projecting from a rigid bar, with successive edges protruding farther from the axis of the bar. Figure 25 illustrates the parts and nomenclature of the broach. Most broaching machines are driven hydraulically and are of the pull or push type.

The maximum force an internal pull broach can withstand without damage is given by

$$P = \frac{A_y F_y}{s} \text{ lb} \tag{57}$$

where A_y = minimum tool selection, in.^{2}
F_y = tensile yield strength of tool steel, psi
s = safety factor

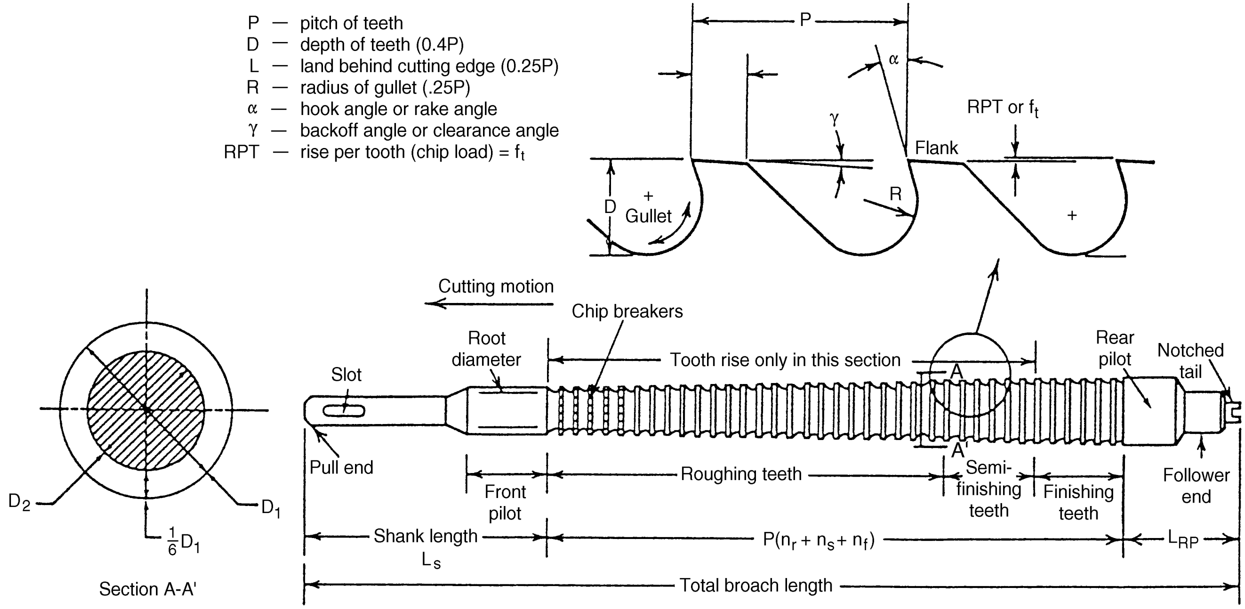


Fig. 25 Standard broach part and nomenclature.

The maximum push force is determined by the minimum tool diameter (D_y), the length of the broach (L), and the minimum compressive yield strength (F_y). The ratio L/D_y should be less than 25 so that the tool will not bend under load. The maximum allowable pushing force is given by

$$P = \frac{A_y F_y}{s} \quad \text{lb} \tag{58}$$

where F_y is minimum compressive yield strength.

If L/D_y ratio is greater than 25 (long broach), the *Tool and Manufacturing Engineers Handbook* gives the following formula:

$$P = \frac{5.6 \times 10^7 D_r^4}{sL^2} \quad \text{lb} \tag{59}$$

D_r and L are given in inches.

Alignment charts were developed for determining metal removal rate (MRR) and motor power in surface broaching. Figures 26 and 27 show the application of these charts for either English or metric units.

Broaching speeds are relatively low, seldom exceeding 50 fpm, but, because a surface is usually completed in one stroke, the productivity is high.

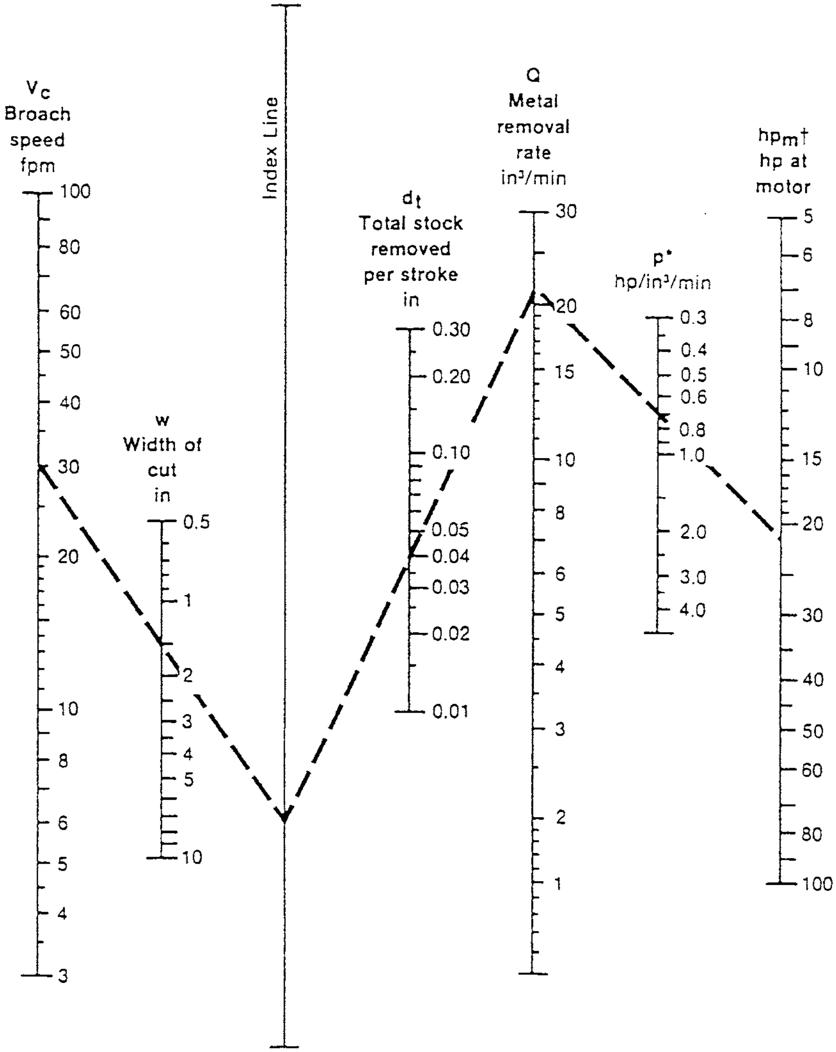
12 SHAPING, PLANING, AND SLOTTING

The shaping and planing operations generate surfaces with a single-point tool by a combination of a reciprocating motion along one axis and a feed motion normal to that axis (Fig. 28). Slots and limited inclined surfaces can also be produced. In shaping, the tool is mounted on a reciprocating ram and the table is fed at each stroke of the ram. Planers handle large, heavy workpieces. In planing, the workpiece reciprocates and the feed increment is provided by moving the tool at each reciprocation. To reduce the lost time on the return stroke, they are provided with a quick-return mechanism. For mechanically driven shapers, the ratio of cutting time to return stroke averages 3:2, and for hydraulic shapers the ratio is 2:1. The average cutting speed may be determined by the following formula:

$$CS = \frac{LN}{12C} \quad \text{fpm} \tag{60}$$

where N = strokes per minute
L = stroke length, in.
C = cutting time ratio, cutting time divided by total time

For mechanically driven shapers, the cutting speed reduces to



Example:

Material: Cast iron — HSS tools

Chipload 0.005 in/tooth

V_c = 30 fpm   w = 1.5 in

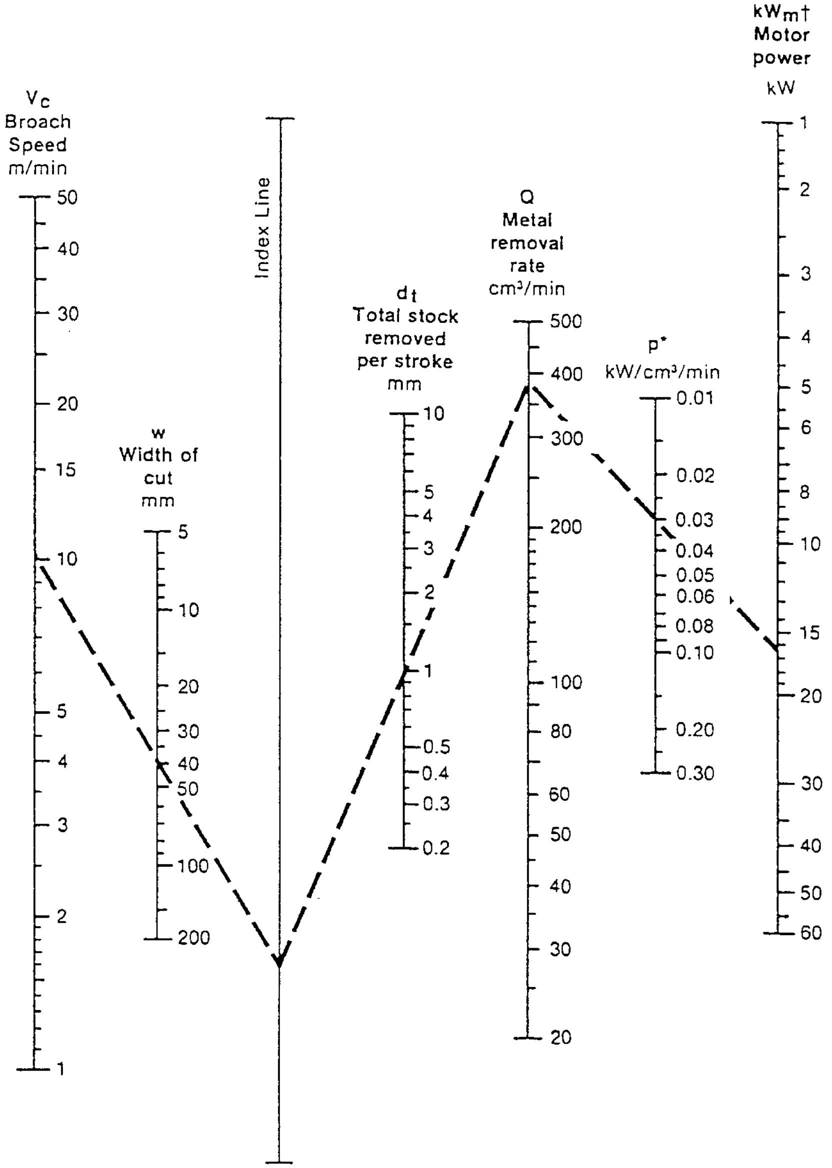
d_t = 0.040 in   Q = 22 in³/min

P = 0.7 hp/in³/min hp_m = 22 hp

$$Q = 12\ V_c \times w \times d_t\ \text{in}^3/\text{min}$$

$$hp_m = \frac{Q \times P}{E} = \frac{Q \times P}{0.7}$$

Fig. 26 Alignment chart for determining metal removal rate and motor horsepower in surface broaching with high-speed steel broaching tools—English units.



Example:

Material: Cast iron — HSS tools

Chipload 0.13 mm/tooth

V_c = 10 m/min w = 38 mm

d_t = 1 mm Q = 380 cm³/min

P = 0.03 kW/cm³/min P_m = 16.3 kW

$$Q = V_c \times w \times d_t \text{ cm}^3/\text{min}$$

$$P_m = \frac{Q \times P}{E} = \frac{Q \times P}{0.7}$$

Fig. 27 Alignment chart for determining metal removal rate and motor power in surface broaching with high-speed steel broaching tools—metric units.

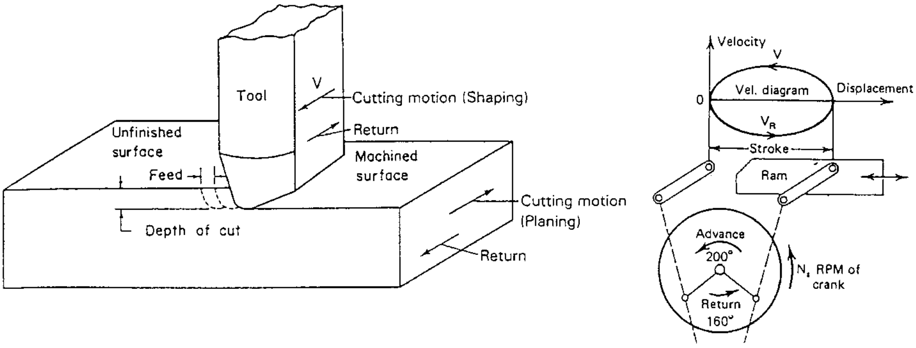


Fig. 28 Basic relationships of tool motion, feed, and depth of cut in shaping and planing.

$$CS = \frac{LN}{7.2} \quad \text{fpm} \tag{61}$$

or

$$CS = \frac{L_1 N}{600} \quad \text{m/min} \tag{62}$$

where L_1 is the stroke length in millimeters. For hydraulically driven shapers,

$$CS = \frac{LN}{8} \quad \text{fpm} \tag{63}$$

or

$$CS = \frac{L_1 N}{666.7} \quad \text{m/min} \tag{64}$$

The time T required to machine a workpiece of width W (in.) is calculated by

$$T = \frac{W}{N \times f} \quad \text{min} \tag{65}$$

where f = feed, in. per stroke

The number of strokes (S) required to complete a job is then

$$S = \frac{W}{f} \tag{66}$$

The power required can be approximated by

$$HP_c = Kdf(CS) \tag{67}$$

where d = depth of cut, in.
CS = cutting speed, fpm
K = cutting constant, for medium cast iron, 3; free-cutting steel, 6; and bronze, 1.5 or

$$HP_c = 12f \times d \times CS \times HP_\mu$$

$$F_c = \frac{33{,}000\ HP_c}{CS}$$

13 SAWING, SHEARING, AND CUTTING OFF

Saws are among the most common of machine tools, even though the surfaces they produce often require further finishing operations. Saws have two general areas of applications: contouring and cutting off. There are three basic types of saws: hacksaw, circular, and band saw.

The *reciprocating power hacksaw* machines can be classified as either positive or uniform-pressure feeds. Most of the new machines are equipped with a quick-return action to reduce idle time.

The machining time required to cut a workpiece of width W in. is calculated as follows:

$$T = \frac{W}{fN} \quad \text{min} \tag{68}$$

where F = feed, in./stroke
N = number of strokes per min

Circular saws are made of three types: metal saws, steel friction disks, and abrasive disks. Solid metal saws are limited in size, not exceeding 16 in. in diameter. Large circular saws have either replaceable inserted teeth or segmented-type blades. The machining time required to cut a workpiece of width W in. is calculated as follows:

$$T = \frac{W}{f_t nN} \quad \text{min} \tag{69}$$

where f_t = feed per tooth
n = number of teeth
N = rpm

Steel friction disks operate at high peripheral speeds ranging from 18,000–25,000 fpm (90–125 m/sec). The heat of friction quickly softens a path through the part. The disk, which is sometimes provided with teeth or notches, pulls and ejects the softened metal. About 0.5 min are required to cut through a 24-in. I-beam.

Abrasive disks are mainly aluminum oxide grains or silicon carbide grains bonded together. They will cut ferrous or nonferrous metals. The finish and accuracy is better than steel friction blades, but they are limited in size compared to steel friction blades.

Band saw blades are of the continuous type. Band sawing can be used for cutting and contouring. Band-sawing machines operate with speeds that range from 50–1500 fpm. The time required to cut a workpiece of width W in. can be calculated as follows:

$$T = \frac{W}{12 f_t n V} \quad \text{min} \tag{70}$$

where f_t = feed, in. per tooth
n = number of teeth per in.
V = cutting speed, fpm

Cutting can also be achieved by band-friction cutting blades with a surface speed up to 15,000 fpm. Other band tools include band filing, diamond bands, abrasive bands, spiral bands, and special-purpose bands.

14 MACHINING PLASTICS

Most plastics are readily formed, but some machining may be required. Plastic's properties vary widely. The general characteristics that affect their machinability are discussed below.

First, all plastics are poor heat conductors. Consequently, little of the heat that results from chip formation will be conducted away through the material or carried away in the chips. As a result, cutting tools run very hot and may fail more rapidly than when cutting metal. Carbide tools frequently are more economical to use than HSS tools if cuts are of moderately long duration or if high-speed cutting is to be done.

Second, because considerable heat and high temperatures do develop at the point of cutting, thermoplastics tend to soften, swell, and bind or clog the cutting tool. Thermosetting plastics give less trouble in this regard.

Third, cutting tools should be kept very sharp at all times. Drilling is best done by means of straight-flute drills or by "dubbing" the cutting edge of a regular twist drill to produce a zero rake angle. Rotary files and burrs, saws, and milling cutters should be run at high speeds in order to improve cooling, but with feed carefully adjusted to avoid jamming the gullets. In some cases, coolants can be used advantageously if they do not discolor the plastic or cause gumming. Water, soluble oil and water, and weak solutions of sodium silicate in water are used. In turning and milling plastics, diamond tools provide the best accuracy, surface finish, and uniformity of finish. Surface speeds of 500–600 fpm with feeds of 0.002–0.005 in. are typical.

Fourth, filled and laminated plastics usually are quite abrasive and may produce a fine dust that may be a health hazard.

15 GRINDING, ABRASIVE MACHINING, AND FINISHING

Abrasive machining is the basic process in which chips are removed by very small edges of abrasive particles, usually synthetic. In many cases, the abrasive particles are bonded into wheels of different shapes and sizes. When wheels are used mainly to produce accurate dimensions and smooth surfaces, the process is called *grinding.* When the primary objective is rapid metal removal to obtain a desired shape or approximate dimensions, it is termed *abrasive machining.* When fine abrasive particles are used to produce very smooth surfaces and to improve the metallurgical structure of the surface, the process is called *finishing.*

15.1 Abrasives

Aluminum oxide (Al_2O_3), usually synthetic, performs best on carbon and alloy steels, annealed malleable iron, hard bronze, and similar metals. Al_2O_3 wheels are not used in grinding very hard materials, such as tungsten carbide, because the grains will get dull prior to fracture. Common trade names for aluminum oxide abrasives are *Alundum* and *Aloxite.*

Silicon carbide (SiC), usually synthetic, crystals are very hard, being about 9.5 on the Moh's scale, where diamond hardness is 10. SiC crystals are brittle, which limits their use. Silicon carbide wheels are recommended for materials of low tensile strength, such as cast iron, brass, stone, rubber, leather, and cemented carbides.

Cubic boron nitride (CBN) is the second-hardest natural or manmade substance. It is good for grinding hard and tough-hardened tool-and-die steels.

Diamonds may be classified as natural or synthetic. Commercial diamonds are now manufactured in high, medium, and low impact strength.

Grain Size

To have uniform cutting action, abrasive grains are graded into various sizes, indicated by the numbers 4–600. The number indicates the number of openings per linear inch in a standard screen through which most of the particles of a particular size would pass. Grain sizes from 4–24 are termed coarse; 30–60, medium; and 70–600, fine. Fine grains produce smoother surfaces than coarse ones but cannot remove as much metal.

Bonding materials have the following effects on the grinding process: (1) they determine the strength of the wheel and its maximum speed; (2) they determine whether the wheel is rigid or flexible; and (3) they determine the force available to pry the particles loose. If only a small force is needed to release the grains, the wheel is said to be soft. Hard wheels are recommended for soft materials and soft wheels for hard materials. The bonding materials used are vitrified, silicate, rubber, resinoid, shellac, and oxychloride.

Structure or Grain Spacing

Structure relates to the spacing of the abrasive grain. Soft, ductile materials require a wide spacing to accommodate the relatively large chips. A fine finish requires a wheel with a close spacing. Figure 29 shows the standard system of grinding wheels as adopted by the American National Standards Institute.

Speeds

Wheel speed depends on the wheel type, bonding material, and operating conditions. Wheel speeds range between 4500 and 18,000 sfpm (22.86 and 27.9 m/s). 5500 sfpm (27.9 m/s) is generally recommended as best for all disk-grinding operations. Work speeds depend on type of material, grinding operation, and machine rigidity. Work speeds range between 15 and 200 fpm.

Feeds

Cross feed depends on the width of grinding wheel. For rough grinding, the range is one-half to three-quarters of the width of the wheel. Finer feed is required for finishing, and it ranges between one-tenth and one-third of the width of the wheel. A cross feed between 0.125 and 0.250 in. is generally recommended.

Depth of Cut

Rough-grinding conditions will dictate the maximum depth of cut. In the finishing operation, the depth of cut is usually small, 0.0002–0.001 in. (0.005–0.025 mm). Good surface finish and close tolerance can be achieved by ‘‘sparking out’’ or letting the wheel run over the workpiece without increasing the depth of cut till sparks die out. The *grinding ratio* (G-ratio) refers to the ratio of the cubic inches of stock removed to the cubic inches of grinding wheel worn away. G-ratio is important in calculating grinding and abrasive machining cost, which may be calculated by the following formula:

$$C = \frac{C_a}{G} + \frac{L}{tq} \tag{71}$$

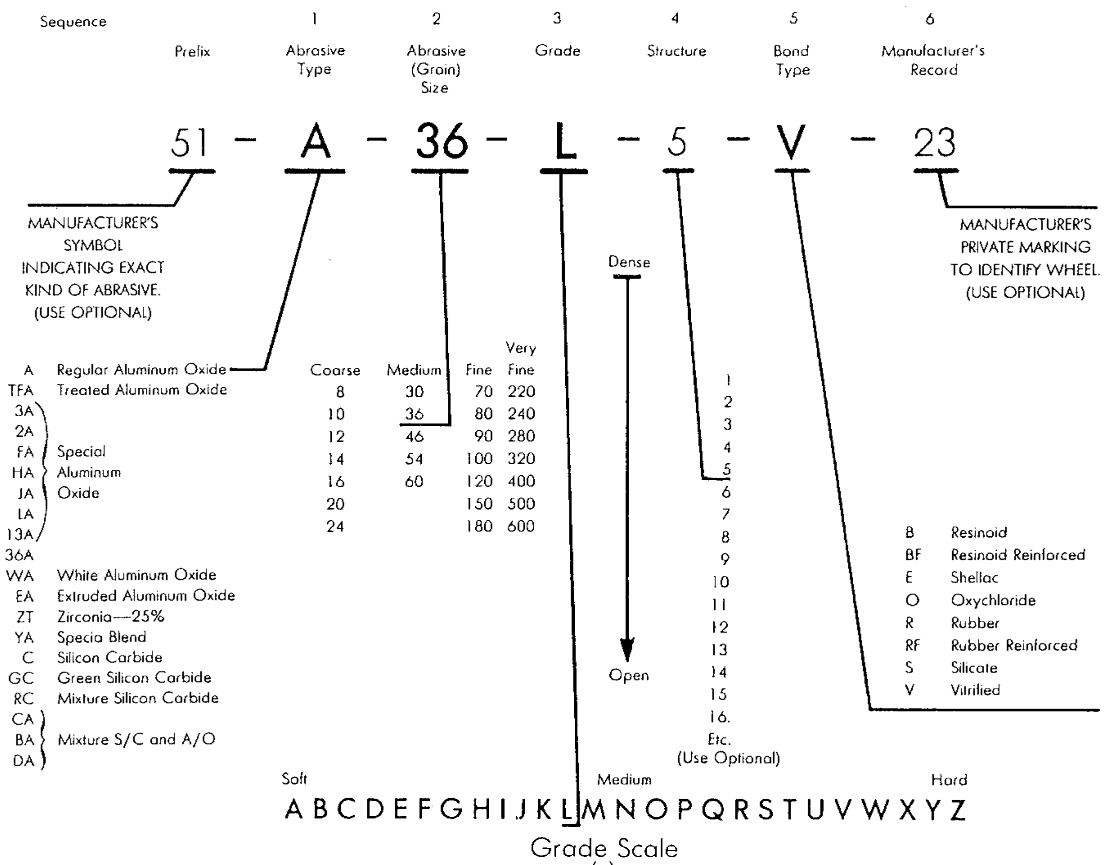
where C = specific cost of removing a cu in. of material
C_a = cost of abrasive, \$/in.^{3}
G = grinding ratio
L = labor and overhead charge, \$/hr
q = machining rate, in.^{3}/hr
t = fraction of time the wheel is in contact with workpiece

Power Requirement

$$\text{Power} = (u)(\text{MRR}) = F_c \times R \times 2\pi N$$

$$\text{MRR} = \text{material removal rate} = d \times w \times v$$

where d = depth of cut
w = width of cut
v = work speed
u = specific energy for surface grinding. Table 14 gives the approximate specific energy requirement for certain metals.
R = radius of wheel
N = rev/unit time


Sequence
Prefix
1
Abrasive Type
2
Abrasive (Grain) Size
3
Grade
4
Structure
5
Bond Type
6
Manufacturer's Record
51 - A - 36 - L - 5 - V - 23
MANUFACTURER'S SYMBOL INDICATING EXACT KIND OF ABRASIVE. (USE OPTIONAL)
A Regular Aluminum Oxide
TFA Treated Aluminum Oxide
3A
2A
FA
HA
JA
LA
13A
Special Aluminum Oxide
36A
WA White Aluminum Oxide
EA Extruded Aluminum Oxide
ZT Zirconia—25%
YA Specia Blend
C Silicon Carbide
GC Green Silicon Carbide
RC Mixture Silicon Carbide
CA
BA
DA
Mixture S/C and A/O
Coarse
8
10
12
14
16
20
24
Medium
30
36
46
54
60
Fine
70
80
90
100
120
150
180
Very Fine
220
240
280
320
400
500
600
Dense
Open
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16.
Etc.
(Use Optional)
B Resinoid
BF Resinoid Reinforced
E Shellac
O Oxychloride
R Rubber
RF Rubber Reinforced
S Silicate
V Vitrified
MANUFACTURER'S PRIVATE MARKING TO IDENTIFY WHEEL. (USE OPTIONAL)
Soft
Medium
Hard
A B C D E F G H I J K L M N O P Q R S T U V W X Y Z
Grade Scale

(a)

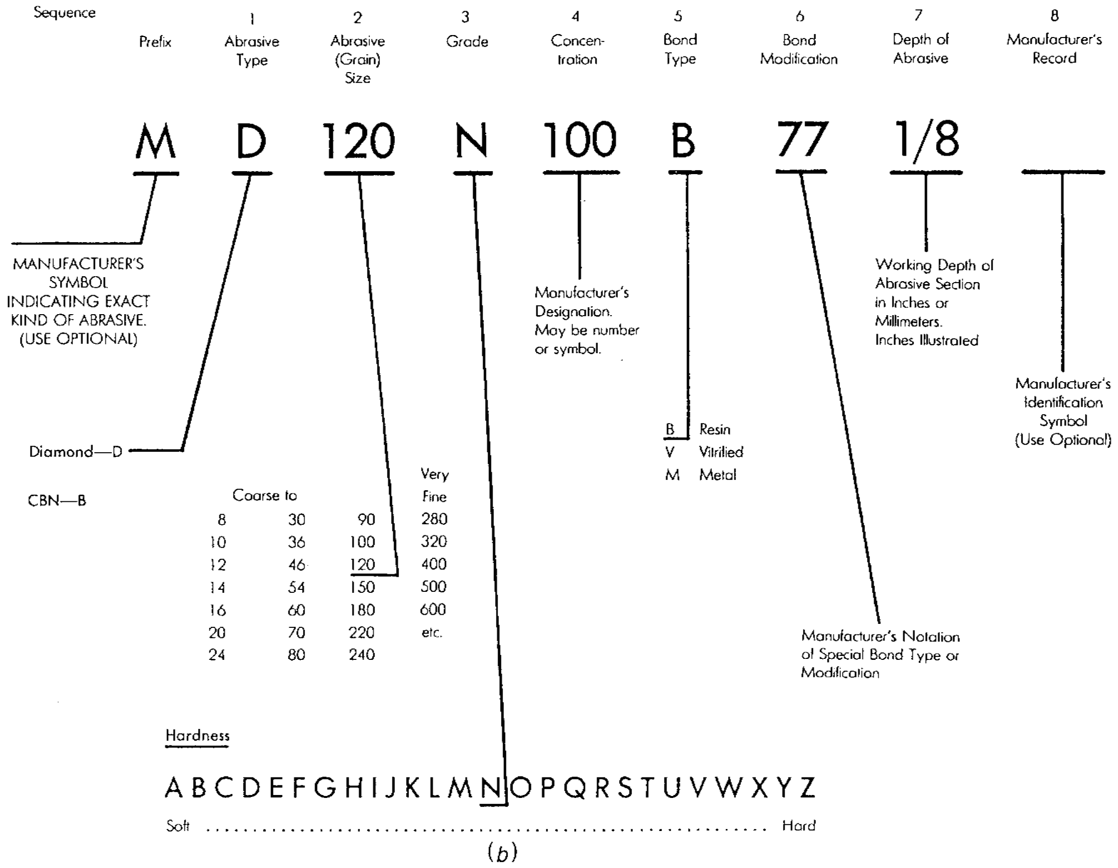


Fig. 29 Standard systems for grinding wheels. (*a*) aluminum oxide, silicon carbide; (*b*) diamond, CBN.

Table 14 Approximate Specific Energy Required for Surface Grinding

Workpiece Material	Hardness	hp (in.^{3}/min)	W/(mm^3/sec)
Aluminum	150 HB	3–10	8–27
Steel	(110–220) HB	6–24	16–66
Cast iron	(140–250) HB	5–22	14–60
Titanium alloy	300 HB	6–20	16–55
Tool steel	62–67 HRC	7–30	19–82

15.2 Temperature

Temperature rise affects the surface properties and causes residual stresses on the workpiece. It is related to process variables by the following relation:

$$\text{temperature rise} \propto D^{1/4} d^{3/4} \left(\frac{V}{v}\right)^{1/2} \qquad (72)$$

where D = wheel diameter
V = wheel speed

Grinding Fluids

Grinding fluids are water-base emulsions for general guiding and oils for thread and gear grinding. Advantages include:

1. Machining hard materials > RC50.
2. Fine surface finish, 10–80 μin. (0.25–2 μm).
3. Accurate dimensions and close tolerances I.0002 in. (I.005 mm) can be easily achieved.
4. Grinding pressure is light.

Machines

Grinding and abrasive machines include

1. Surface grinders, reciprocating or rotating table
2. Cylindrical grinders, work between centers, centerless, crankshaft, thread and gear form work, and internal and other special applications
3. Jig grinders
4. Tool and cutter grinders
5. Snagging, foundry rough work
6. Cutting off and profiling
7. Abrasive grinding, belt, disk and loose grit
8. Mass media, barrel tumbling, and vibratory

Ultrasonic Machining

In ultrasonic machining, material is removed from the workpiece by microchipping or erosion through high-velocity bombardment by abrasive particles, in the form of a slurry, through the action of an ultrasonic transducer. It is used for machining hard and brittle materials and can produce very small and accurate holes 0.015 in. (0.4 mm).

Surface Finishing

Finishing processes produce an extra-fine surface finish; in addition, tool marks are removed and very close tolerances are achieved. Some of these processes follow.

Honing is a low-velocity abrading process. It uses fine abrasive stones to remove very small amounts of metals usually left from previous grinding processes. The amount of metal removed is usually less than 0.005 in. (0.13 mm). Because of low cutting speeds, heat and pressure are minimized, resulting in excellent sizing and metallurgical control.

Lapping is an abrasive surface-finishing process wherein fine abrasive particles are charged in some sort of a vehicle, such as grease, oil, or water, and are embedded into a soft material, called a *lap.* Metal laps must be softer than the work and are usually made of close-grained gray cast iron. Other materials, such as steel, copper, and wood, are used where cast iron is not suitable. As the charged lap is rubbed against a surface, small amounts of material are removed from the harder surface. The amount of material removed is usually less than 0.001 in. (0.03 mm).

Superfinishing is a surface-improving process that removes undesirable fragmentation, leaving a base of solid crystalline metal. It uses fine abrasive stones, like honing, but differs in the type of motion. Very rapid, short strokes, very light pressure, and low-viscosity lubricant–coolant are used in superfinishing. It is essentially a finishing process and not a dimensional one, and can be superimposed on other finishing operations.

Buffing

Buffing wheels are made from a variety of soft materials. The most widely used is muslin, but flannel, canvas, sisal, and heavy paper are used for special applications. Buffing is usually divided into two operations: cutting down and coloring. The first is used to smooth the surface and the second to produce a high luster. The abrasives used are extremely fine powders of aluminum oxide, tripoli (an amorphous silicon), crushed flint or quartz, silicon carbide, and red rouge (iron oxide). Buffing speeds range between 6,000 and 12,000 fpm.

Electropolishing is the reverse of electroplating; that is, the work is the anode instead of the cathode and metal is removed rather than added. The electrolyte attacks projections on the workpiece surface at a higher rate, thus producing a smooth surface.

16 NONTRADITIONAL MACHINING

Nontraditional, or nonconventional, machining processes are material-removal processes that have recently emerged or are new to the user. They have been grouped for discussion here according to their primary energy mode; that is, mechanical, electrical, thermal, or chemical, as shown in Table 15.

Nontraditional processes provide manufacturing engineers with additional choices or alternatives to be applied where conventional processes are not satisfactory, such as when

Table 15 Current Commercially Available Nontraditional Material Removal Processes

Mechanical		Electrical		Thermal		Chemical	
AFM	Abrasive flow machining	ECD	Electrochemical deburring	EBM	Electron-beam machining	CHM	Chemical machining: chemical milling, chemical blanking
AJM	Abrasive jet machining	ECDG	Electrochemical discharge grinding	EDG	Electrical discharge grinding		
HDM	Hydrodynamic machining						
LSG	Low-stress grinding	ECG	Electrochemical grinding	EDM	Electrical discharge machining	ELP	Electropolish
RUM	Rotary ultrasonic machining	ECH	Electrochemical honing			PCM	Photochemical machining
		ECM	Electrochemical machining	EDS	Electrical discharge sawing	TCM	Thermochemical machining (or TEM, thermal energy method)
TAM	Thermally assisted machining	ECP	Electrochemical polishing	EDWC	Electrical discharge wire cutting		
		ECS	Electrochemical sharpening				
TFM	Total form machining	ECT	Electrochemical turning	LBM	Laser-beam machining		
USM	Ultrasonic machining	ES	Electro-stream™	LBT	Laser-beam torch		
WJM	Water-jet machining	STEM™	Shaped tube electrolytic machining	PBM	Plasma-beam machining		

- Shapes and dimensions are complex or very small
- Hardness of material is very high (>400 HB)
- Tolerances are tight and very fine surface finish is desired
- Temperature rise and residual stresses must be avoided
- Cost and production time must be reduced

Figure 30 and Table 16 demonstrate the relationships among the conventional and the nontraditional machining processes with respect to surface roughness, dimensional tolerance, and metal-removal rate.

The *Machinery Handbook*[6] is an excellent reference for nontraditional machining processes, values, ranges, and limitations.

16.1 Abrasive Flow Machining

Abrasive flow machining (AFM) is the removal of material by a viscous, abrasive medium flowing, under pressure, through or across a workpiece. Figure 31 contains a schematic presentation of the AFM process. Generally, the putty-like medium is extruded through or over the workpiece with motion usually in both directions. Aluminum oxide, silicon carbide, boron carbide, or diamond abrasives are used. The movement of the abrasive matrix erodes away burrs and sharp corners and polishes the part.

16.2 Abrasive Jet Machining

Abrasive jet machining (AJM) is the removal of material through the action of a focused, high-velocity stream of fine grit or powder-loaded gas. The gas should be dry, clean, and under modest pressure. Figure 32 shows a schematic of the AJM process. The mixing chamber sometimes uses a vibrator to promote a uniform flow of grit. The hard nozzle is directed close to the workpiece at a slight angle.

16.3 Hydrodynamic Machining

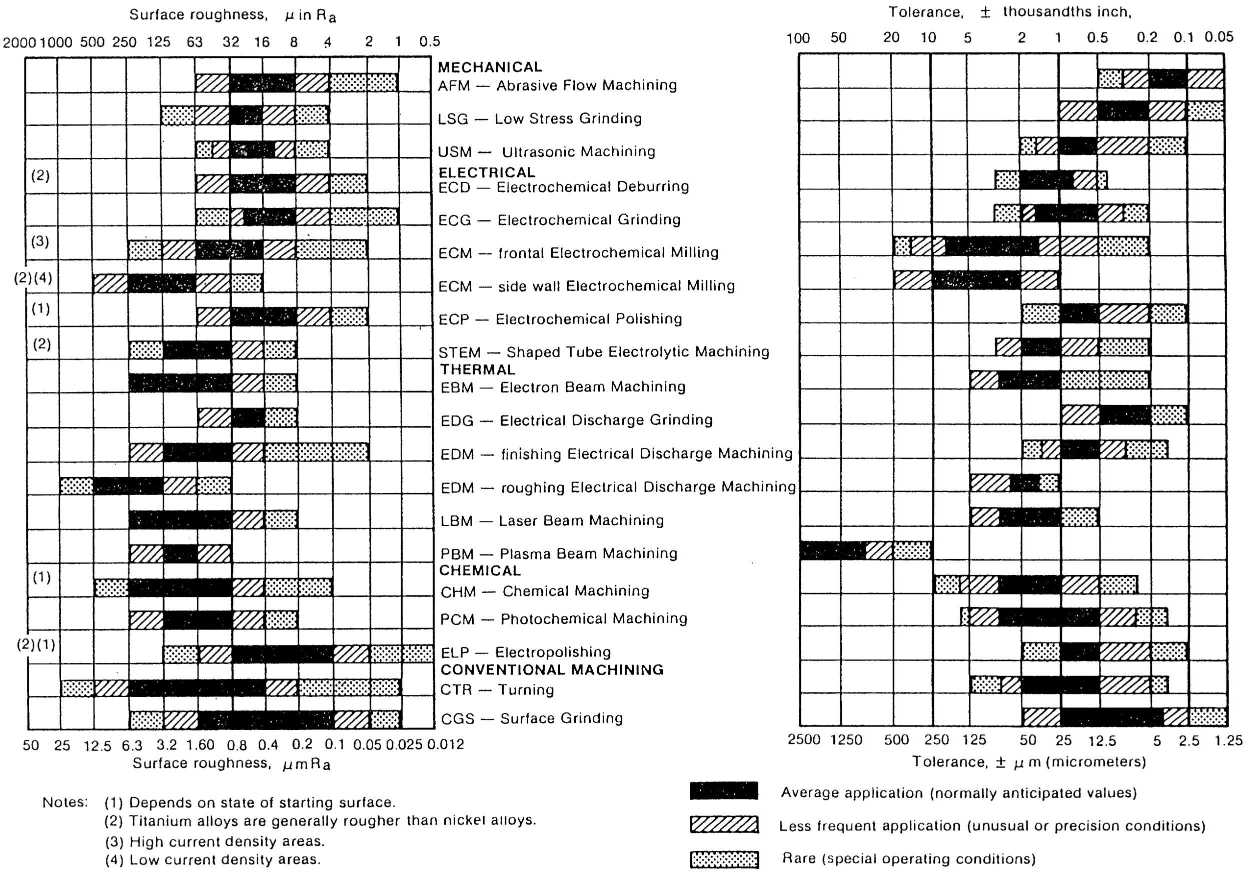
Hydrodynamic machining (HDM) removes material by the stroking of high-velocity fluid against the workpiece. The jet of fluid is propelled at speeds up to Mach 3. Figure 33 shows a schematic of the HDM operation.

16.4 Low-Stress Grinding

Low-stress grinding (LSG) is an abrasive material-removal process that leaves a low-magnitude, generally compressive residual stress in the surface of the workpiece. Figure 34 shows a schematic of the LSG process. The thermal effects from conventional grinding can produce high tensile stress in the workpiece surface. The process parameter guidelines can be applied to any of the grinding modes: surface, cylindrical, centerless, internal, and so on.

16.5 Thermally Assisted Machining

Thermally assisted machining (TAM) is the addition of significant amounts of heat to the workpiece immediately prior to single-point cutting so that the material is softened but the strength of the tool bit is unimpaired (Fig. 35). While resistive heating and induction heating offer possibilities, the plasma arc has a core temperature of 14,500°F (8000°C) and a surface temperature of 6500°F



Notes: (1) Depends on state of starting surface.
(2) Titanium alloys are generally rougher than nickel alloys.
(3) High current density areas.
(4) Low current density areas.

Fig. 30 Typical surface roughness and tolerances produced by nontraditional machining.

Table 16 Material Removal Rates and Dimensional Tolerances

					Accuracy ±		
	Maximum Rate of Material Removal $in.^3/min$	Typical Power Consumption $hp/in.^3/min$	Cutting Speed fpm	Penetration Rate per Minute in.	Attainable in.	At Maximum Material Removal Rate in.	Typical Machine Input hp
Process	cm^3/min	$kW/cm^3/min$	m/min	mm	mm	mm	kW
Conventional turning	200	1	250	—	0.0002	0.005	30
	3300	0.046	76	—	0.005	0.13	22
Conventional grinding	50	10	10	—	0.0001	0.002	25
	820	0.46	3	—	0.0025	0.05	20
CHM	30	—	—	0.001	0.0005	0.003	—
	490	—	—	0.025	0.013	0.075	—
PBM	10	20	50	10	0.02	0.1	200
	164	0.91	15	254	0.5	2.54	150
ECG	2	2	0.25	—	0.0002	0.0025	4
	33	0.019	0.08	—	0.005	0.063	3
ECM	1	160	—	0.5	0.0005	0.006	200
	16.4	7.28	—	12.7	0.013	0.15	150
EDM	0.3	40	—	0.5	0.00015	0.002	15
	4.9	1.82	—	12.7	0.004	0.05	11
USM	0.05	200	—	0.02	0.0002	0.0015	15
	0.82	9.10	—	0.50	0.005	0.040	11
EBM	0.0005	10,000	200	6	0.0002	0.002	10
	0.0082	455	60	150	0.005	0.050	7.5
LBM	0.0003	60,000	—	4	0.0005	0.005	20
	0.0049	2,731	—	102	0.013	0.13	15

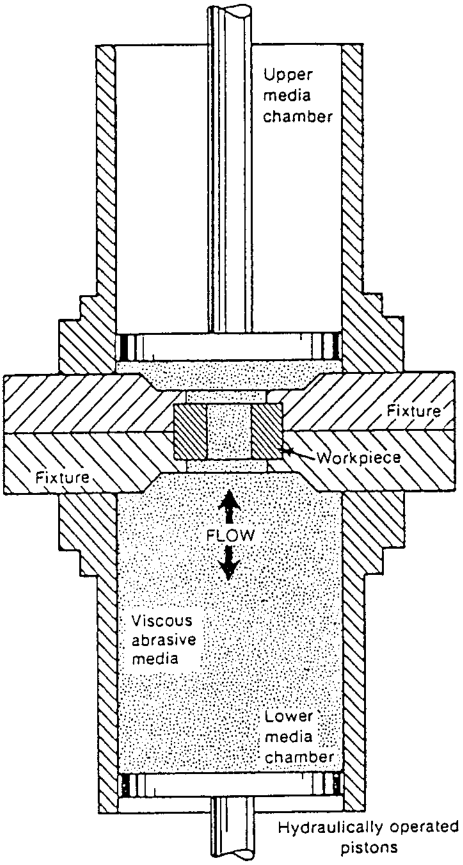


Fig. 31 Abrasive flow machining.

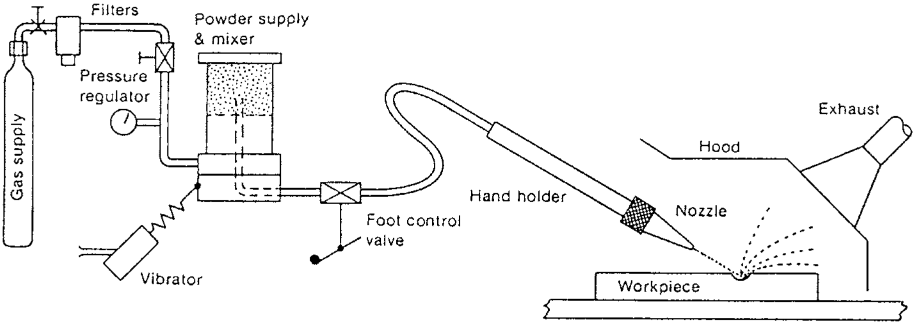


Fig. 32 Abrasive jet machining.

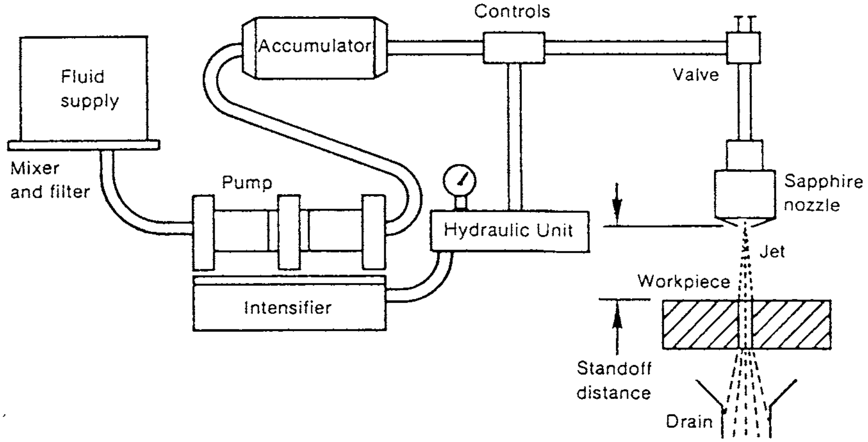


Fig. 33 Hydrodynamic machining.

(3600°C). The torch can produce 2000°F (1100°C) in the workpiece in approximately one-quarter revolution of the workpiece between the point of application of the torch and the cutting tool.

16.6 Electromechanical Machining

Electromechanical machining (EMM) is a process in which the metal removal is effected in a conventional manner except that the workpiece is electrochemically polarized. When the applied voltage and the electrolytic solution are controlled, the surface of the workpiece can be changed to achieve the characteristics suitable for the machining operation.

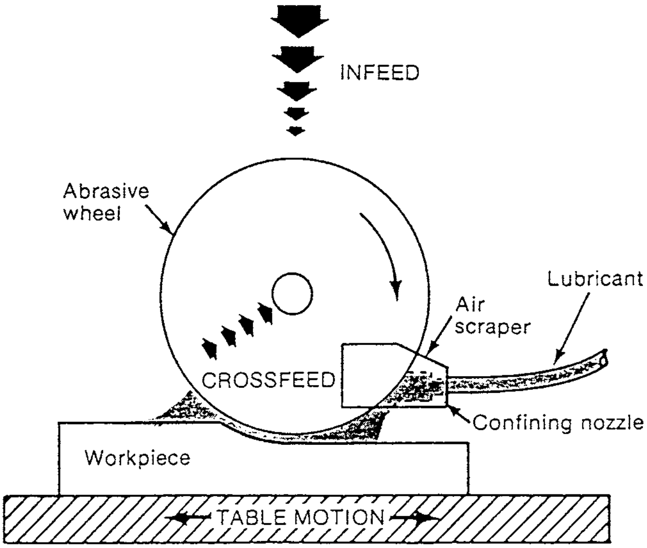


Fig. 34 Low-stress grinding.

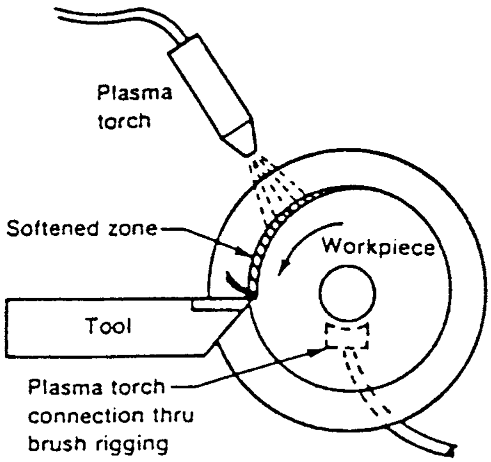


Fig. 35 Thermally assisted machining.

16.7 Total Form Machining

Total form machining (TFM) is a process in which an abrasive master abrades its full three-dimensional shape into the workpiece by the application of force while a full-circle, orbiting motion is applied to the workpiece via the worktable (Fig. 36). The cutting master is advanced into the work until the desired depth of cut is achieved. Uniformity of cutting is promoted by the fluid that continuously transports the abraded particles out of the working gap. Adjustment of the orbiting cam drive controls the precision of the overcut from the cutting master. Cutting action takes place simultaneously over the full surface of abrasive contact.

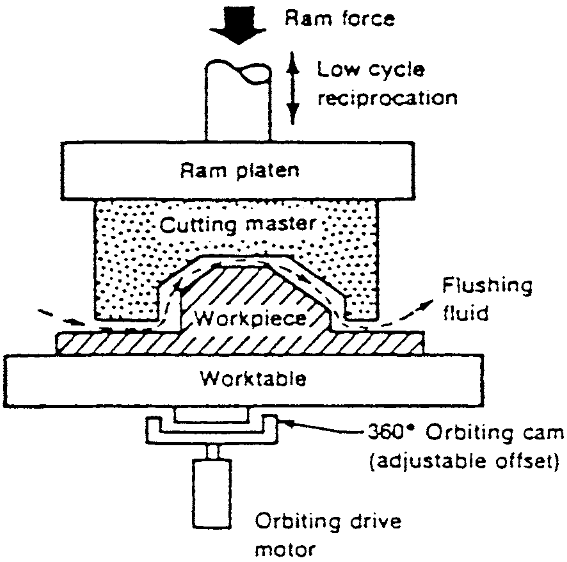


Fig. 36 Total form machining.

16.8 Ultrasonic Machining

Ultrasonic machining (USM) is the removal of material by the abrading action of a grit-loaded liquid slurry circulating between the workpiece and a tool vibrating perpendicular to the workface at a frequency above the audible range (Fig. 37). A high-frequency power source activates a stack of magnetostrictive material, which produces a low-amplitude vibration of the toolholder. This motion is transmitted under light pressure to the slurry, which abrades the workpiece into a conjugate image of the tool form. A constant flow of slurry (usually cooled) is necessary to carry away the chips from the workface. The process is sometimes called *ultrasonic abrasive machining* (UAM) or *impact machining.*

A prime variation of USM is the addition of ultrasonic vibration to a rotating tool—usually a diamond-plated drill. *Rotary ultrasonic machining* (RUM) substantially increases the drilling efficiency. A piezoelectric device built into the rotating head provides the needed vibration. Milling, drilling, turning, threading, and grinding-type operations are performed with RUM.

16.9 Water-Jet Machining

Water-jet machining (WJM) is low-pressure hydrodynamic machining. The pressure range for WJM is an order of magnitude below that used in HDM. There are two versions of WJM: one for mining, tunneling, and large-pipe cleaning

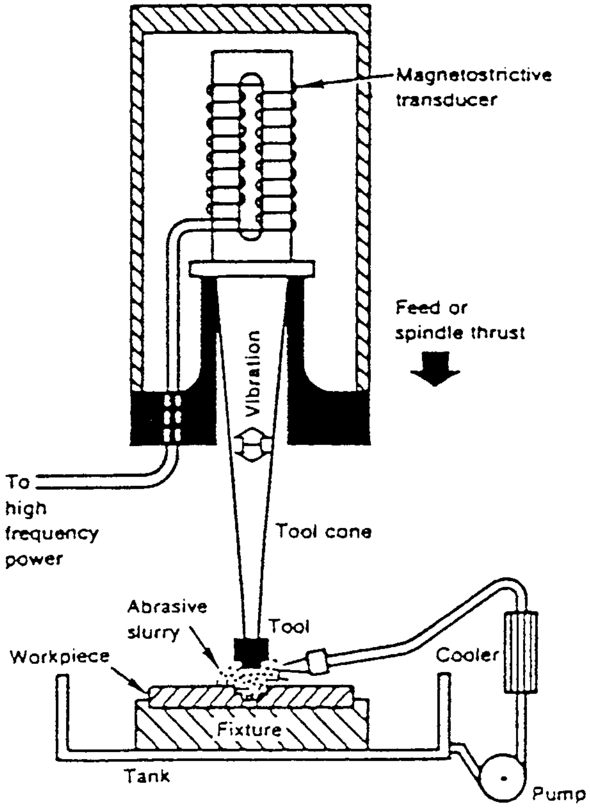


Fig. 37 Ultrasonic machining.

that operates in the region from 250–1000 psi (1.7–6.9 Mpa); and one for smaller parts and production shop situations that uses pressures below 250 psi (1.7 Mpa).

The first version, or high-pressure range, is characterized by use of a pumped water supply with hoses and nozzles that generally are hand-directed. In the second version, more production-oriented and controlled equipment, such as that shown in Fig. 38, is involved. In some instances, abrasives are added to the fluid flow to promote rapid cutting. Single or multiple-nozzle approaches to the workpiece depend on the size and number of parts per load. The principle is that WJM is high-volume, not high-pressure.

16.10 Electrochemical Deburring

Electrochemical deburring (ECD) is a special version of ECM (Fig. 39). ECD was developed to remove burrs and fins or to round sharp corners. Anodic dissolution occurs on the workpiece burrs in the presence of a closely placed cathodic tool whose configuration matches the burred edge. Normally, only a small portion of the cathode is electrically exposed, so a maximum concentration of the electrolytic action is attained. The electrolyte flow usually is arranged to carry away any burrs that may break loose from the workpiece during the cycle. Voltages are low, current densities are high, electrolyte flow rate is modest, and electrolyte types are similar to those used for ECM. The electrode (tool) is stationary, so equipment is simpler than that used for ECM. Cycle time is short for deburring. Longer cycle time produces a natural radiusing action.

16.11 Electrochemical Discharge Grinding

Electrochemical discharge grinding (ECDG) combines the features of both electrochemical and electrical discharge methods of material removal (Fig. 40).

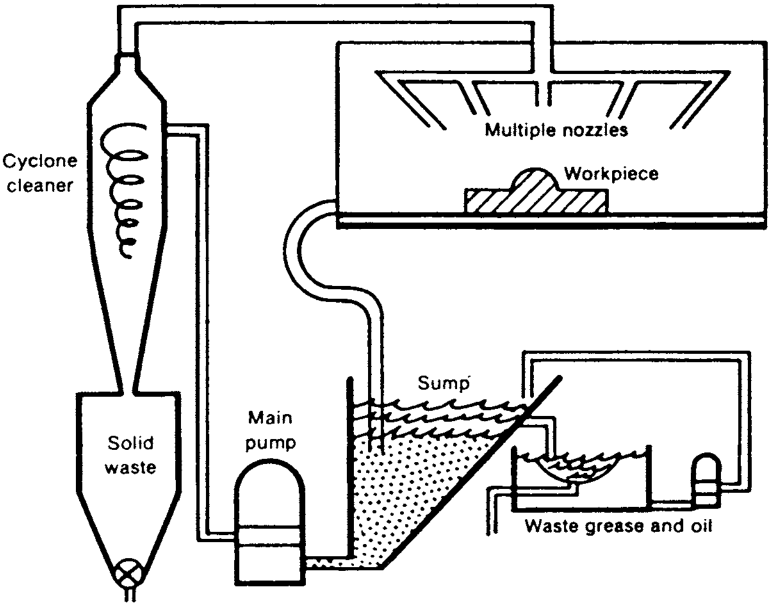


Fig. 38 Water-jet machining.

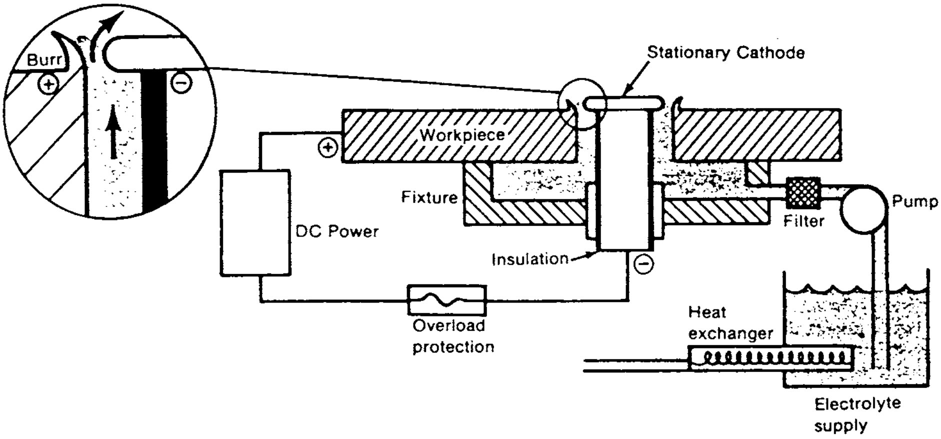


Fig. 39 Electrochemical deburring.

ECDG has the arrangement and electrolytes of electrochemical grinding (ECG), but uses a graphite wheel without abrasive grains. The random spark discharge is generated through the insulating oxide film on the workpiece by the power generated in an ac source or by a pulsating dc source. The principal material removal comes from the electrolytic action of the low-level dc voltages. The spark discharges erode the anodic films to allow the electrolytic action to continue.

16.12 Electrochemical Grinding

Electrochemical grinding (ECG) is a special form of electrochemical machining in which the conductive workpiece material is dissolved by anodic action, and any resulting films are removed by a rotating, conductive, abrasive wheel (Fig. 41). The abrasive grains protruding from the wheel form the insulating electrical gap between the wheel and the workpiece. This gap must be filled with electrolyte at all times. The conductive wheel uses conventional abrasives—aluminum oxide (because it is nonconductive) or diamond (for intricate shapes)—but lasts substantially longer than wheels used in conventional grinding. The reason for this is that the bulk of material removal (95–98%) occurs by deplating, while

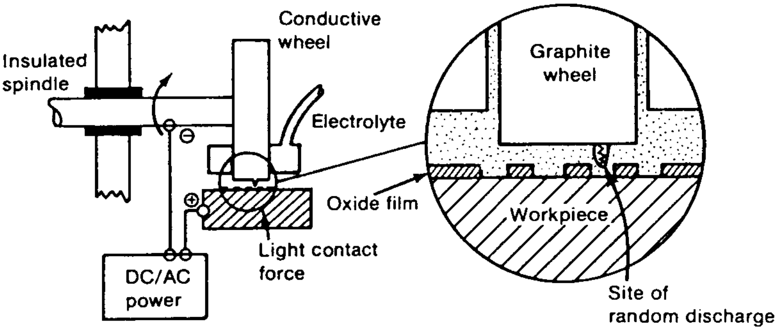


Fig. 40 Electrochemical discharge grinding.

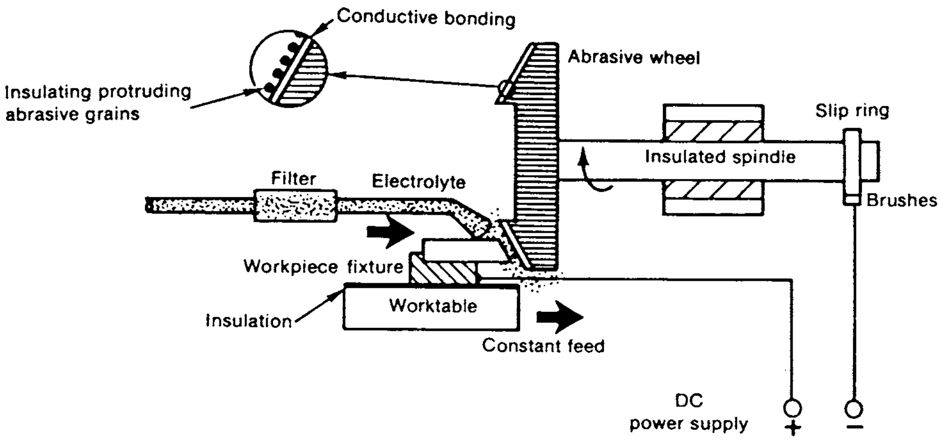


Fig. 41 Electrochemical grinding.

only a small amount (2–5%) occurs by abrasive mechanical action. Maximum wheel contact arc lengths are about $\frac{3}{4}$–1 in. (19–25 mm) to prevent overheating the electrolyte. The fastest material removal is obtained by using the highest attainable current densities without boiling the electrolyte. The corrosive salts used as electrolytes should be filtered and flow rate should be controlled for the best process control.

16.13 Electrochemical Honing

Electrochemical honing (ECH) is the removal of material by anodic dissolution combined with mechanical abrasion from a rotating and reciprocating abrasive stone (carried on a spindle, which is the cathode) separated from the workpiece by a rapidly flowing electrolyte (Fig. 42). The principal material removal action comes from electrolytic dissolution. The abrasive stones are used to maintain size and to clean the surfaces to expose fresh metal to the electrolyte action. The small electrical gap is maintained by the nonconducting stones that are bonded to the expandable arbor with cement. The cement must be compatible with the electrolyte and the low dc voltage. The mechanical honing action uses materials, speeds, and pressures typical of conventional honing.

16.14 Electrochemical Machining

Electrochemical machining (ECM) is the removal of electrically conductive material by anodic dissolution in a rapidly flowing electrolyte, which separates the workpiece from a shaped electrode (Fig. 43). The filtered electrolyte is pumped under pressure and at controlled temperature to bring a controlled-conductivity fluid into the narrow gap of the cutting area. The shape imposed on the workpiece is nearly a mirror or conjugate image of the shape of the cathodic electrode. The electrode is advanced into the workpiece at a constant feed rate that exactly matches the rate of dissolution of the work material. Electrochemical machining is basically the reverse of electroplating.

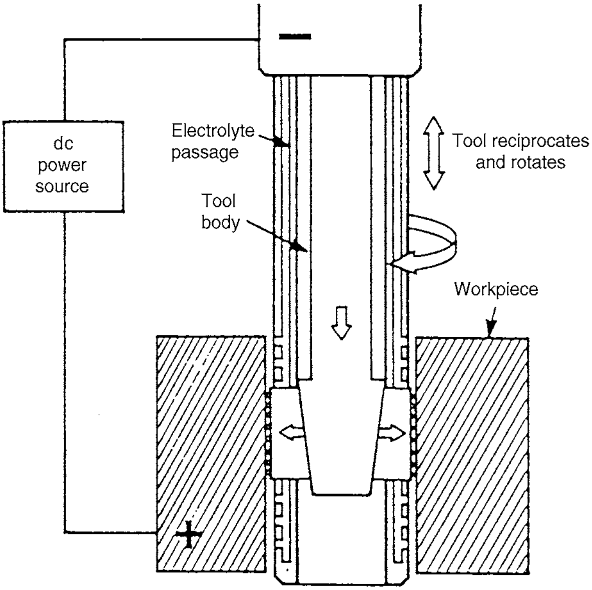


Fig. 42 Electrochemical honing.

Calculation of Metal Removal and Feed Rates in ECM

$$\text{current } I = \frac{V}{R} \text{ amp}$$

$$\text{resistance } R = \frac{g \times r}{A}$$

where g = length of gap (cm)
r = electrolyte resistivity

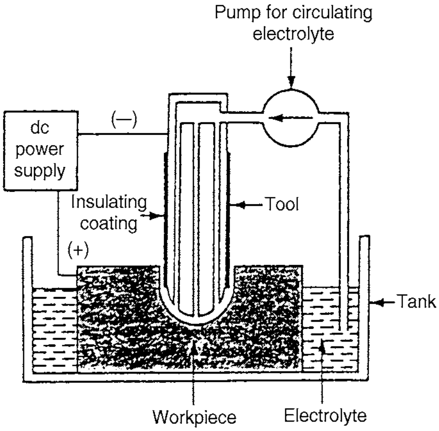


Fig. 43 Electrochemical machining.

A = area of current path (cm^2)
V = voltage
R = resistance

$$\text{current density } S = \frac{I}{A} = \frac{V}{r \times g} \quad \text{amp/cm}^2$$

The amount of material deposited or dissolved is proportional to the quantity of electricity passed (current × time).

$$\text{amount of material} = C \times I \times t$$

where C = constant
t = time, sec

The amount removed or deposited by one faraday (96,500 coulombs = 96,500 amp–sec) is 1 gram-equivalent weight (G)

$$G = \frac{N}{n} \quad \text{(for 1 faraday)}$$

where N = atomic weight
n = valence

$$\text{volume of metal removed} = \frac{I \times t}{96{,}500} \times \frac{N}{n} \times \frac{1}{d} \times h$$

where d = density, g/cm^3
h = current efficiency

$$\text{specific removal rate } s = \frac{N}{n} \times \frac{1}{96{,}500} \times h \quad \text{cm}^3\text{/amp–sec}$$

$$\text{cathode feed rate } F = S \times s \quad \text{cm/sec.}$$

16.15 Electrochemical Polishing

Electrochemical polishing (ECP) is a special form of electrochemical machining arranged for cutting or polishing a workpiece (Fig. 44). Polishing parameters are similar in range to those for cutting, but without the feed motion. ECP generally uses a larger gap and a lower current density than does ECM. This requires modestly higher voltages. (In contrast, electropolishing (ELP) uses still lower current densities, lower electrolyte flow, and more remote electrodes.)

16.16 Electrochemical Sharpening

Electrochemical sharpening (ECS) is a special form of electrochemical machining arranged to accomplish sharpening or polishing by hand (Fig. 45). A portable power pack and electrolyte reservoir supply a finger-held electrode with a small current and flow. The fixed gap incorporated on the several styles of shaped

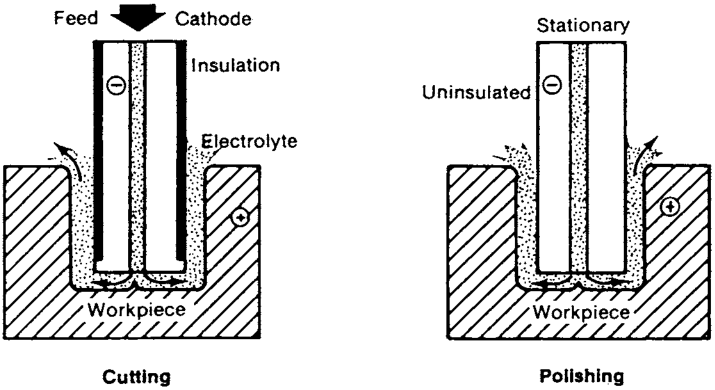


Fig. 44 Electrochemical polishing.

electrodes controls the flow rate. A suction tube picks up the used electrolyte for recirculation after filtration.

16.17 Electrochemical Turning

Electrochemical turning (ECT) is a special form of electrochemical machining designed to accommodate rotating workpieces (Fig. 46). The rotation provides additional accuracy but complicates the equipment with the method of introducing the high currents to the rotating part. Electrolyte control may also be complicated because rotating seals are needed to direct the flow properly. Otherwise, the parameters and considerations of electrochemical machining apply equally to the turning mode.

16.18 Electro-Stream

Electro-stream (ES) is a special version of electrochemical machining adapted for drilling very small holes using high voltages and acid electrolytes (see Fig. 47). The voltages are more than 10 times those employed in ECM or STEM, so special provisions for containment and protection are required. The tool is a

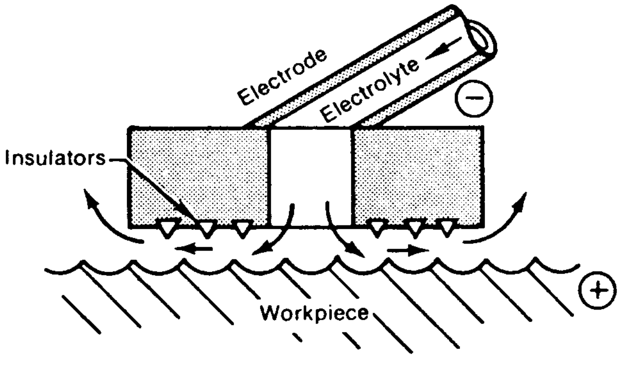


Fig. 45 Electrochemical sharpening.

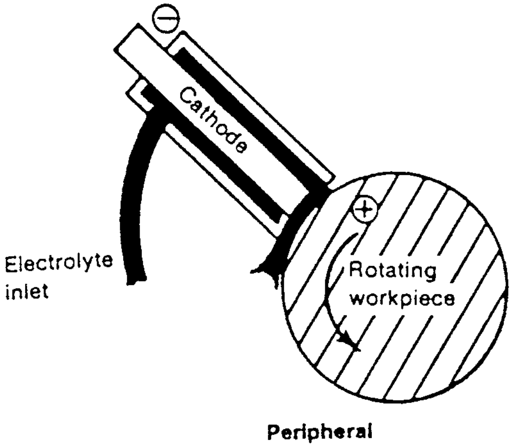


Fig. 46 Electrochemical turning.

drawn-glass nozzle, 0.001–0.002 in. smaller than the desired hole size. An electrode inside the nozzle or the manifold ensures electrical contact with the acid. Multiple-hole drilling is achieved successfully by ES.

16.19 Shaped-Tube Electrolytic Machining

Shaped-tube electrolytic machining (STEM™) is a specialized ECM technique for "drilling" small, deep holes by using acid electrolytes (Fig. 48). Acid is used so that the dissolved metal will go into the solution rather than form a sludge, as is the case with the salt-type electrolytes of ECM. The electrode is a carefully straightened acid-resistant metal tube. The tube is coated with a film of enamel-type insulation. The acid is pressure-fed through the tube and returns via a narrow gap between the tube insulation and the hole wall. The electrode is fed into the workpiece at a rate exactly equal to the rate at which the workpiece

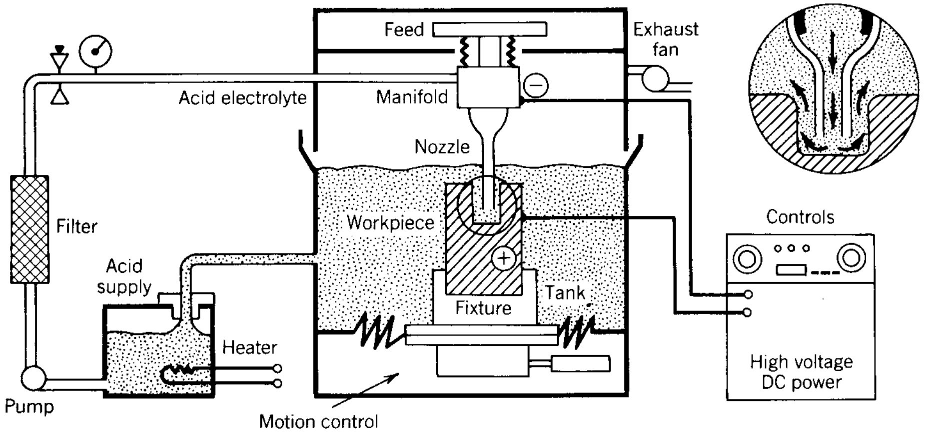


Fig. 47 Electro-stream.

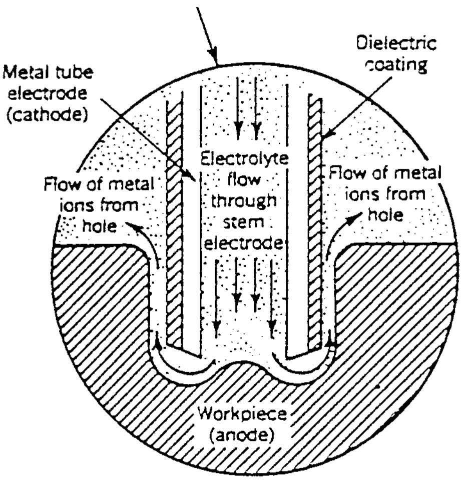


Fig. 48 Shaped-tube electrolytic machining.

material is dissolved. Multiple electrodes, even of varying diameters or shapes, may be used simultaneously. A solution of sulfuric acid is frequently used as the electrolyte when machining nickel alloys. The electrolyte is heated and filtered, and flow monitors control the pressure. Tooling is frequently made of plastics, ceramics, or titanium alloys to withstand the electrified hot acid.

16.20 Electron-Beam Machining

Electron-beam machining (EBM) removes material by melting and vaporizing the workpiece at the point of impingement of a focused stream of high-velocity electrons (Fig. 49). To eliminate scattering of the beam of electrons by contact

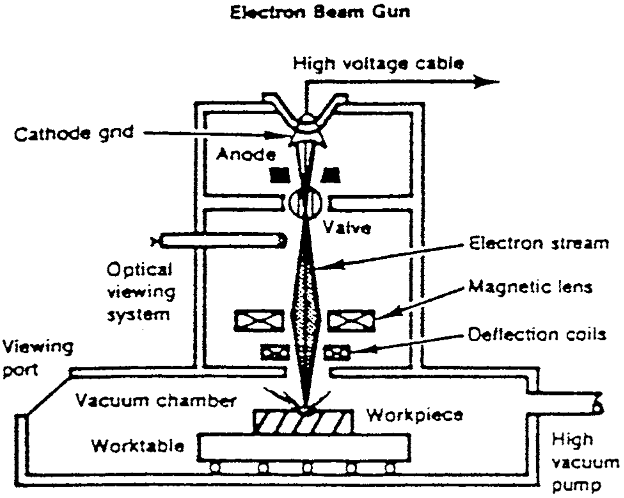


Fig. 49 Electron-beam machining.

with gas molecules, the work is done in a high-vacuum chamber. Electrons emanate from a triode electron-beam gun and are accelerated to three-fourths the speed of light at the anode. The collision of the electrons with the workpiece immediately translates their kinetic energy into thermal energy. The low-inertia beam can be simply controlled by electromagnetic fields. Magnetic lenses focus the electron beam on the workpiece, where a 0.001-in. (0.025-mm) diameter spot can attain an energy density of up to 10^9 W/in.^{2} (1.55×10^8 W/cm^2) to melt and vaporize any material. The extremely fast response time of the beam is an excellent companion for three-dimensional computer control of beam deflection, beam focus, beam intensity, and workpiece motion.

16.21 Electrical Discharge Grinding

Electrical discharge grinding (EDG) is the removal of a conductive material by rapid, repetitive spark discharges between a rotating tool and the workpiece, which are separated by a flowing dielectric fluid (Fig. 50). (EDG is similar to EDM except that the electrode is in the form of a grinding wheel and the current is usually lower.) The spark gap is servocontrolled. The insulated wheel and the worktable are connected to the dc pulse generator. Higher currents produce faster cutting, rougher finishes, and deeper heat-affected zones in the workpiece.

16.22 Electrical Discharge Machining

Electrical discharge machining (EDM) removes electrically conductive material by means of rapid, repetitive spark discharges from a pulsating dc power supply with dielectric flowing between the workpiece and the tool (Fig. 51). The cutting tool (electrode) is made of electrically conductive material, usually carbon. The shaped tool is fed into the workpiece under servocontrol. A spark discharge then breaks down the dielectric fluid. The frequency and energy per spark are set and controlled with a dc power source. The servocontrol maintains a constant gap between the tool and the workpiece while advancing the electrode. The dielectric oil cools and flushes out the vaporized and condensed material while reestablishing insulation in the gap. Material removal rate ranges from 16–245 cm^3/h.

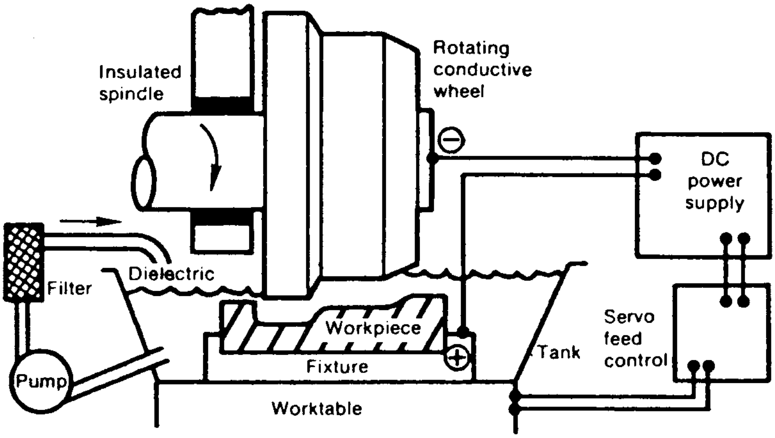


Fig. 50 Electrical discharge grinding.

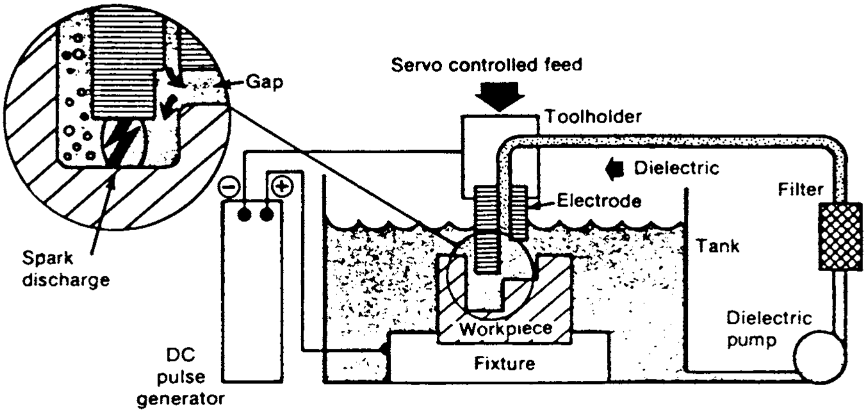


Fig. 51 Electrical discharge machining.

EDM is suitable for cutting materials regardless of their hardness or toughness. Round or irregular-shaped holes 0.002 in. (0.05 mm) diameter can be produced with L/D ratio of 20:1. Narrow slots as small as 0.002–0.010 in. (0.05–0.25 mm) wide are cut by EDM.

16.23 Electrical Discharge Sawing

Electrical discharge sawing (EDS) is a variation of electrical discharge machining (EDM) that combines the motion of either a band saw or a circular disk saw with electrical erosion of the workpiece (Fig. 52). The rapid-moving, untoothed, thin, special steel band or disk is guided into the workpiece by carbide-faced inserts. A kerf only 0.002–0.005 in. (0.050–0.13 mm) wider than the blade or disk is formed as they are fed into the workpiece. Water is used as a cooling quenchant for the tool, swarf, and workpiece. Circular cutting is usually performed under water, thereby reducing noise and fumes. While the work is power-

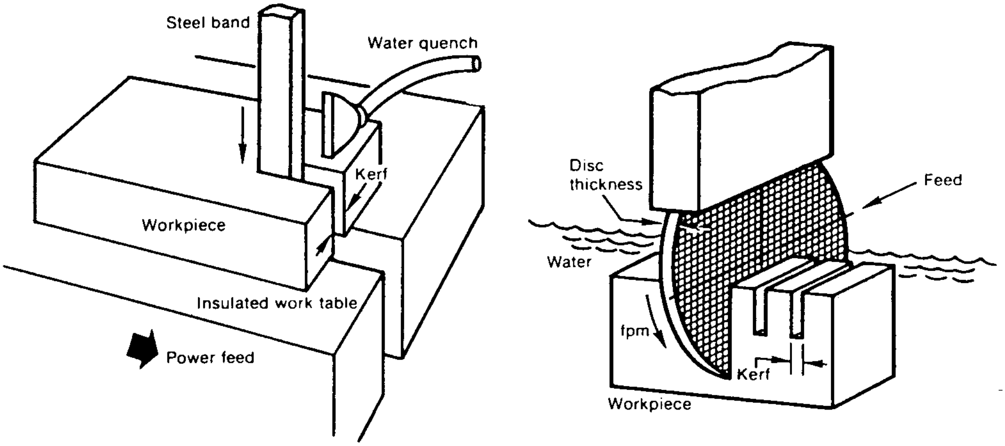


Fig. 52 Electrical discharge sawing.

fed into the band (or the disk into the work), it is not subjected to appreciable forces because the arc does the cutting, so fixturing can be minimal.

16.24 Electrical Discharge Wire Cutting (Traveling Wire)

Electrical discharge wire cutting (EDWC) is a special form of electrical discharge machining wherein the electrode is a continuously moving conductive wire (Fig. 53). EDWC is often called *traveling wire* EDM. A small-diameter tension wire, 0.001–0.012 in. (0.03–0.30 mm), is guided to produce a straight, narrow-kerf size 0.003–0.015 in. (0.075–0.375 mm). Usually, a programmed or numerically controlled motion guides the cutting, while the width of the kerf is maintained by the wire size and discharge controls. The dielectric is oil or deionized water carried into the gap by motion of the wire. Wire EDM is able to cut plates as thick as 12 in. (300 mm) and issued for making dies from hard metals. The wire travels with speed in the range of 6–300 in./min (0.15–8 mm/min). A typical cutting rate is 1 in.^{2} (645 mm^2) of cross-sectional area per hour.

16.25 Laser-Beam Machining

Laser-beam machining (LBM) removes material by melting, ablating, and vaporizing the workpiece at the point of impingement of a highly focused beam of coherent monochromatic light (Fig. 54). Laser is an acronym for "light amplification by stimulated emission of radiation." The electromagnetic radiation operates at wavelengths from the visible to the infrared. The principal lasers used for material removal are the ND:glass (neodymium–glass), the Nd:YAG (neodymium:yttrium-aluminum-garnet), the ruby and the carbon dioxide (CO_2). The last is a gas laser (most frequently used as a torch with an assisting gas—see LBT, laser-beam torch), while others are solid-state lasing materials.

For pulsed operation, the power supply produces short, intense bursts of electricity into the flash lamps, which concentrate their light flux on the lasing material. The resulting energy from the excited atoms is released at a characteristic, constant frequency. The monochromatic light is amplified during successive reflections from the mirrors. The thoroughly collimated light exits through the

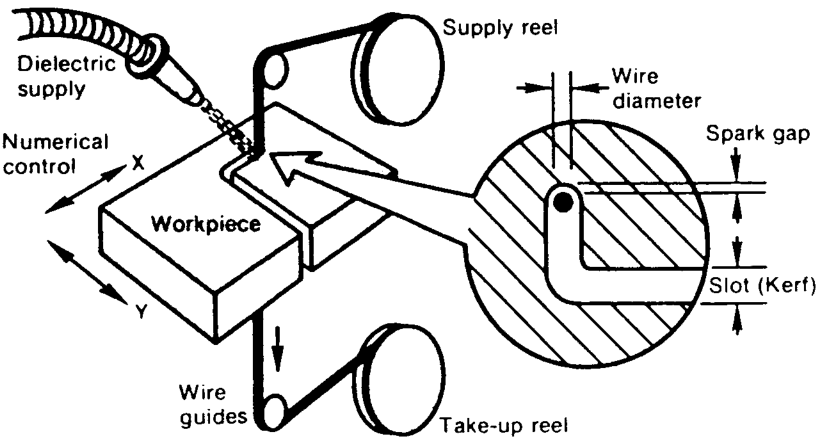


Fig. 53 Electrical discharge wire cutting.

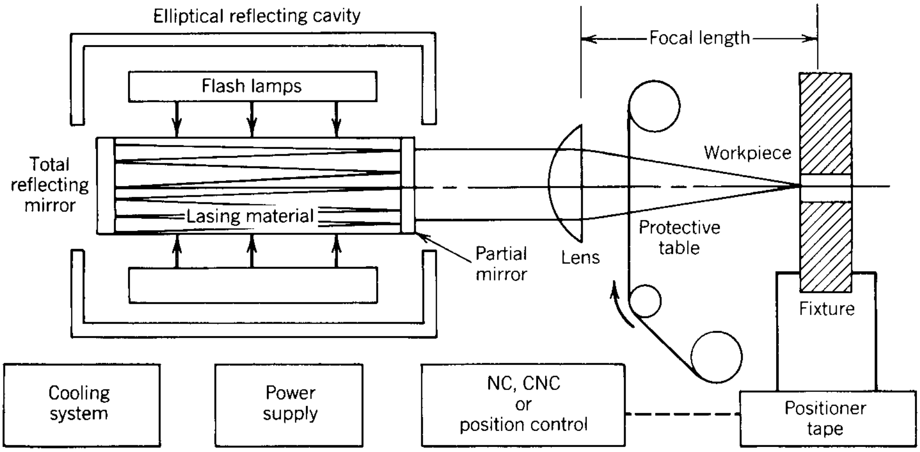


Fig. 54 Laser-beam machining.

partially reflecting mirror to the lens, which focuses it on or just below the surface of the workpiece. The small beam divergence, high peak power, and single frequency provide excellent, small-diameter spots of light with energy densities up to 3×10^{10} W/in.^{2} (4.6×10^9 W/cm^2), which can sublime almost any material. Cutting requires energy densities of 10^7–10^9 W/in.^{2} (1.55×10^6–1.55×10^8 W/cm^2), at which rate the thermal capacity of most materials cannot conduct energy into the body of the workpiece fast enough to prevent melting and vaporization. Some lasers can instantaneously produce 41,000°C (74,000°F). Holes of 0.001 in. (0.025 mm), with depth-to-diameter 50 to 1 are typically produced in various materials by LBM.

16.26 Laser-Beam Torch

Laser-beam torch (LBT) is a process in which material is removed by the simultaneous focusing of a laser beam and a gas stream on the workpiece (see Fig. 55). A continuous-wave (CW) laser or a pulsed laser with more than 100 pulses per second is focused on or slightly below the surface of the workpiece, and the absorbed energy causes localized melting. An oxygen gas stream promotes an exothermic reaction and purges the molten material from the cut. Argon or nitrogen gas is sometimes used to purge the molten material while also protecting the workpiece.

Argon or nitrogen gas is often used when organic or ceramic materials are being cut. Close control of the spot size and the focus on the workpiece surface is required for uniform cutting. The type of gas used has only a modest effect on laser penetrating ability. Typically, short laser pulses with high peak power are used for cutting and welding. The CO_2 laser is the laser most often used for cutting. Thin materials are cut at high rates, $\frac{1}{8}$–$\frac{3}{8}$ in. (3.2–9.5 mm) thickness is a practical limit.

16.27 Plasma-Beam Machining

Plasma-beam machining (PBM) removes material by using a superheated stream of electrically ionized gas (Fig. 56). The 20,000–50,000°F (11,000–28,000°C)

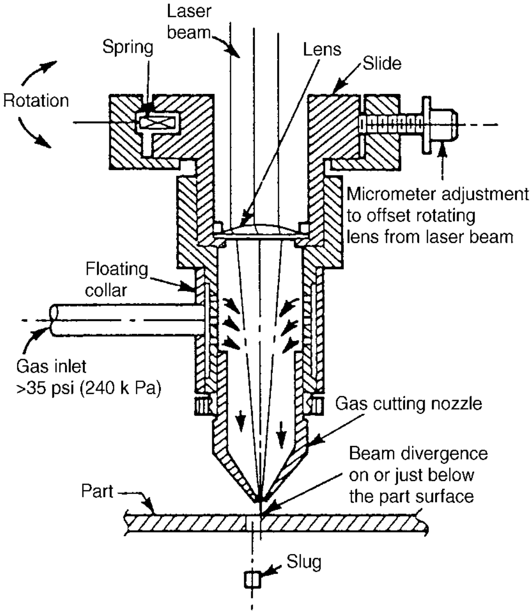


Fig. 55 Laser-beam torch.

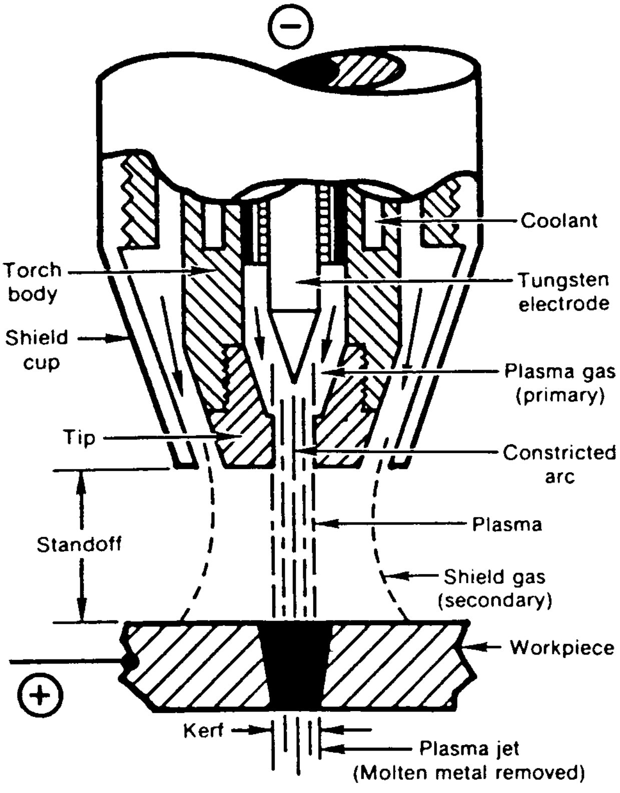


Fig. 56 Plasma-beam machining.

plasma is created inside a water-cooled nozzle by electrically ionizing a suitable gas, such as nitrogen, hydrogen, or argon, or mixtures of these gases. Since the process does not rely on the heat of combustion between the gas and the workpiece material, it can be used on almost any conductive metal. Generally, the arc is transferred to the workpiece, which is made electrically positive. The plasma—a mixture of free electrons, positively charged ions, and neutral atoms—is initiated in a confined, gas-filled chamber by a high-frequency spark. The high-voltage dc power sustains the arc, which exits from the nozzle at near-sonic velocity. The high-velocity gases blow away the molten metal "chips." Dual-flow torches use a secondary gas or water shield to assist in blowing the molten metal out of the kerf, giving a cleaner cut. PBM is sometimes called *plasma-arc cutting* (PAC). PBM can cut plates up to 6.0 in. (152 mm) thick. Kerf width can be as small as 0.06 in. (1.52 mm) in cutting thin plates.

16.28 Chemical Machining: Chemical Milling, Chemical Blanking

Chemical machining (CHM) is the controlled dissolution of a workpiece material by contact with a strong chemical reagent (Fig. 57). The thoroughly cleaned workpiece is covered with a strippable, chemically resistant mask. Areas where chemical action is desired are outlined on the workpiece with the use of a template and then stripped off the mask. The workpiece is then submerged in the chemical reagent to remove material simultaneously from all exposed surfaces. The solution should be stirred or the workpiece should be agitated for more effective and more uniform action. Increasing the temperatures will also expedite the action. The machined workpiece is then washed and rinsed, and the remaining mask is removed. Multiple parts can be maintained simultaneously in the same tank. A wide variety of metals can be chemically machined; however, the practical limitations for depth of cut are 0.25–0.5 in. (6.0–12.0 mm) and typical etching rate is 0.001 in./min (0.025 mm/min).

In chemical blanking, the material is removed by chemical dissolution instead of shearing. The operation is applicable to production of complex shapes in thin sheets of metal.

16.29 Electropolishing

Electropolishing (ELP) is a specialized form of chemical machining that uses an electrical deplating action to enhance the chemical action (Fig. 58). The

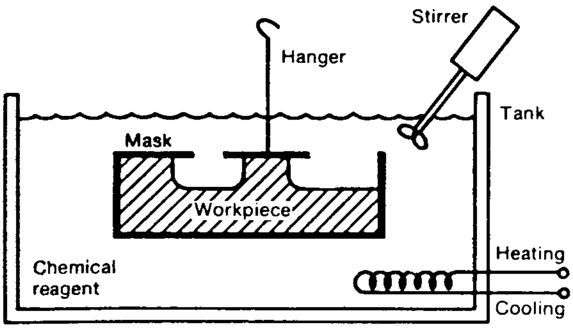


Fig. 57 Chemical machining.

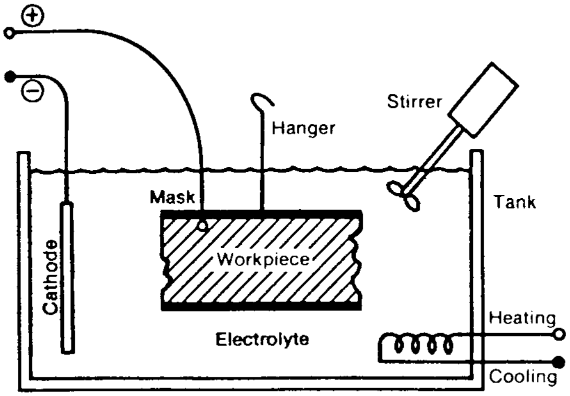


Fig. 58 Electropolishing.

chemical action from the concentrated heavy acids does most of the work, while the electrical action smooths or polishes the irregularities. A metal cathode is connected to a low-voltage, low-amperage dc power source and is installed in the chemical bath near the workpiece. Usually, the cathode is not shaped or conformed to the surface being polished. The cutting action takes place over the entire exposed surface; therefore, a good flow of heated, fresh chemicals is needed in the cutting area to secure uniform finishes. The cutting action will concentrate first on burrs, fins, and sharp corners. Masking, similar to that used with CHM, prevents cutting in unwanted areas. Typical roughness values range from 4–32 μin. (0.1–0.8 μm).

16.30 Photochemical Machining

Photochemical machining (PCM) is a variation of CHM where the chemically resistant mask is applied to the workpiece by a photographic technique (Fig.

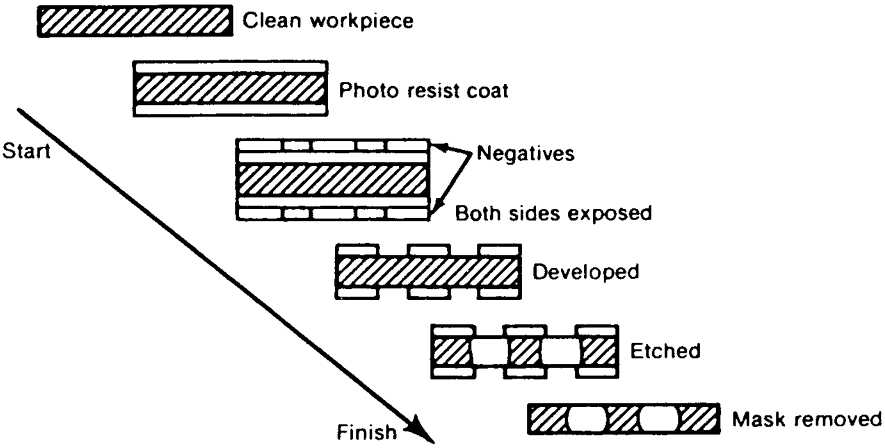


Fig. 59 Photochemical machining.

59). A photographic negative, often a reduced image of an oversize master print (up to 100 ×), is applied to the workpiece and developed. Precise registry of duplicate negatives on each side of the sheet is essential for accurately blanked parts. Immersion or spray etching is used to remove the exposed material. The chemicals used must be active on the workpiece, but inactive against the photoresistant mask. The use of PCM is limited to thin materials—up to $\frac{1}{16}$ in. (1.5 mm).

16.31 Thermochemical Machining

Thermochemical machining (TCM) removes the workpiece material—usually only burrs and fins—by exposure of the workpiece to hot, corrosive gases. The process is sometimes called *combustion machining, thermal deburring,* or *thermal energy method* (TEM). The workpiece is exposed for a very short time to extremely hot gases, which are formed by detonating an explosive mixture. The ignition of the explosive—usually hydrogen or natural gas and oxygen—creates a transient thermal wave that vaporizes the burrs and fins. The main body of the workpiece remains unaffected and relatively cool because of its low surface-to-mass ratio and the shortness of the exposure to high temperatures.

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CHAPTER 31

METAL FORMING, SHAPING, AND CASTING

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1 INTRODUCTION

Metal-forming processes use a remarkable property of metals—their ability to flow plastically in the solid state without concurrent deterioration of properties.

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Handbook of Materials Selection. Edited by Myer Kutz

Moreover, by simply moving the metal to the desired shape, there is little or no waste. Figure 1 shows some of the metal-forming processes. Metal-forming processes are classified into two categories: hot-working processes and cold-working processes.

2 HOT-WORKING PROCESSES

Hot working is defined as the plastic deformation of metals above their recrystallization temperature. Here it is important to note that the crystallization temperature varies greatly with different materials. Lead and tin are hot worked at room temperature, while steels require temperatures of 2000°F (1100°C). Hot working does not necessarily imply high absolute temperatures.

Hot working can produce the following improvements:

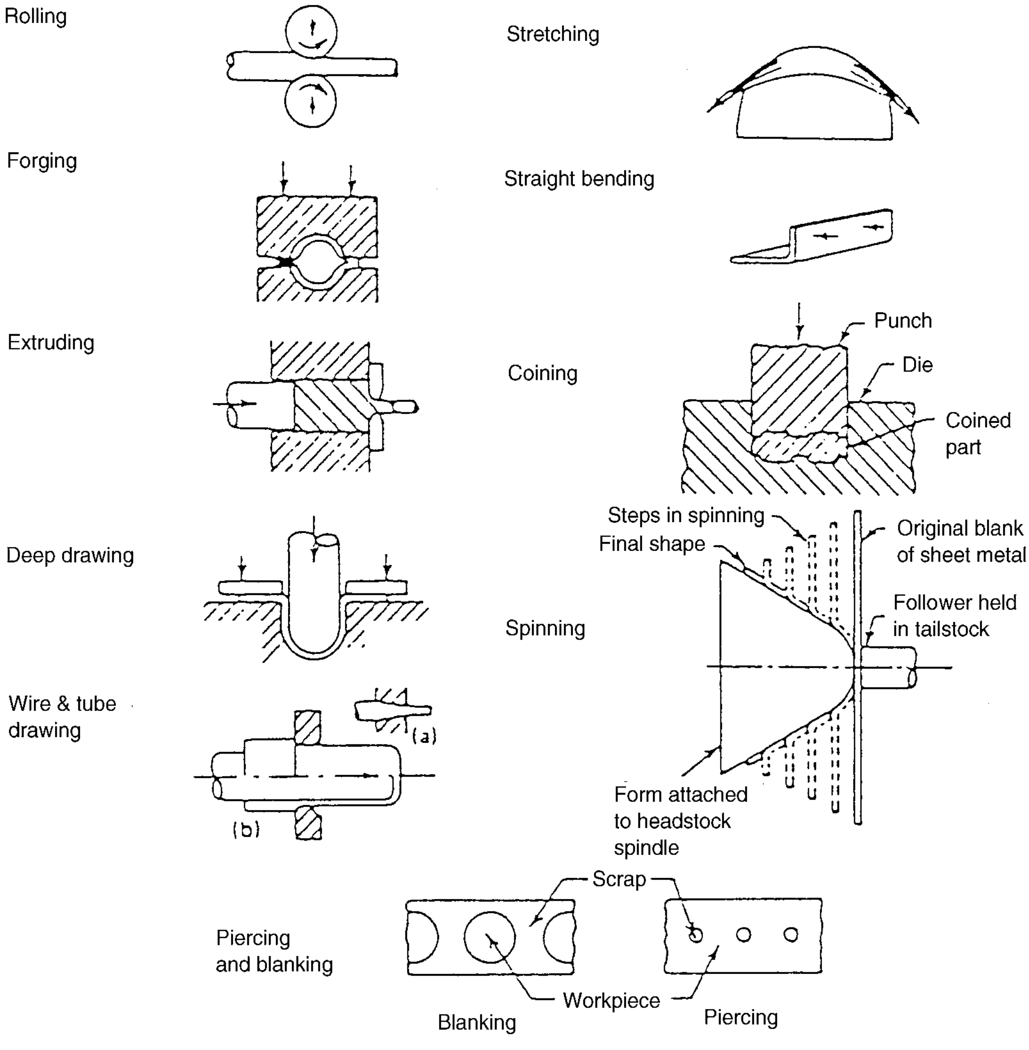

Fig. 1 Metal-forming processes.

1. Production of randomly oriented, spherical-shaped grain structure, which results in a net increase not only in the strength but also in ductility and toughness.
2. The reorientation of inclusions or impurity material in metal. The impurity material often distorts and flows along with the metal.

This material, however, does not recrystallize with the base metal and often produces a fiber structure. Such a structure clearly has directional properties, being stronger in one direction than in another. Moreover, an impurity originally oriented so as to aid crack movement through the metal is often reoriented into a ‘‘crack-arrestor’’ configuration perpendicular to crack propagation.

2.1 Classification of Hot-Working Processes

The most obvious reason for the popularity of hot working is that it provides an attractive means of forming a desired shape. Some of the hot-working processes that are of major importance in modern manufacturing are

1. Rolling
2. Forging
3. Extrusion and upsetting
4. Drawing
5. Spinning
6. Pipe welding
7. Piercing

2.2 Rolling

Hot rolling (Fig. 2) consists of passing heated metal between two rolls that revolve in opposite directions, the space between the rolls being somewhat less than the thickness of the entering metal. Many finished parts, such as hot-rolled structural shapes, are completed entirely by hot rolling. More often, however, hot-rolled products, such as sheets, plates, bars, and strips, serve as input material for other processes, such as cold forming or machining.

In hot rolling, as in all hot working, it is very important that the metal be heated uniformly throughout to the proper temperature, a procedure known as *soaking*. If the temperature is not uniform, the subsequent deformation will also be nonuniform, the hotter exterior flowing in preference to the cooler and, therefore, stronger, interior. Cracking, tearing, and associated problems may result.

Isothermal Rolling

The ordinary rolling of some high-strength metals, such as titanium and stainless steels, particularly in thicknesses below about 0.150 in. (3.8 mm), is difficult because the heat in the sheet is transferred rapidly to the cold and much more massive rolls. This has been overcome by isothermal rolling. Localized heating is accomplished in the area of deformation by the passage of a large electrical current between the rolls, through the sheet. Reductions up to 90% per roll have been achieved. The process usually is restricted to widths below 2 in. (50 mm).

The rolling strip contact length is given by

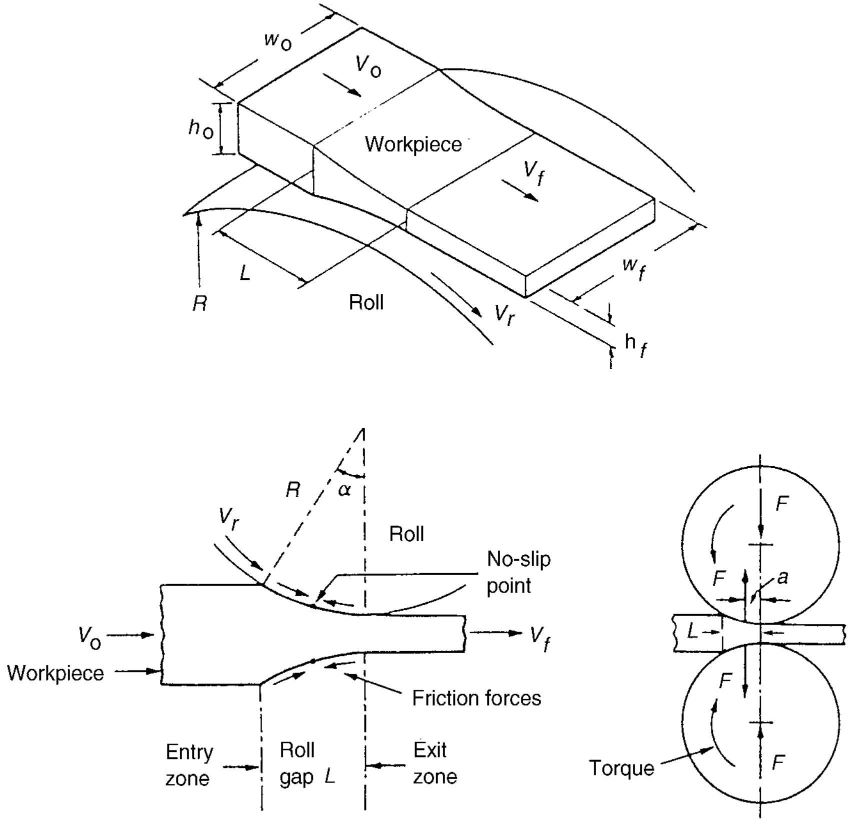

Fig. 2 Hot rolling.

$$L \simeq \sqrt{R(h_0 - h)}$$

where R = roll radius
h_0 = original strip thickness
h = reduced thickness

The roll force F is calculated by

$$F = LwY_{\text{avg}} \tag{1}$$

where w = width
Y_{avg} = average true stress

Figure 3 gives the true stress for different material at the true stress ε. The true stress ε is given by

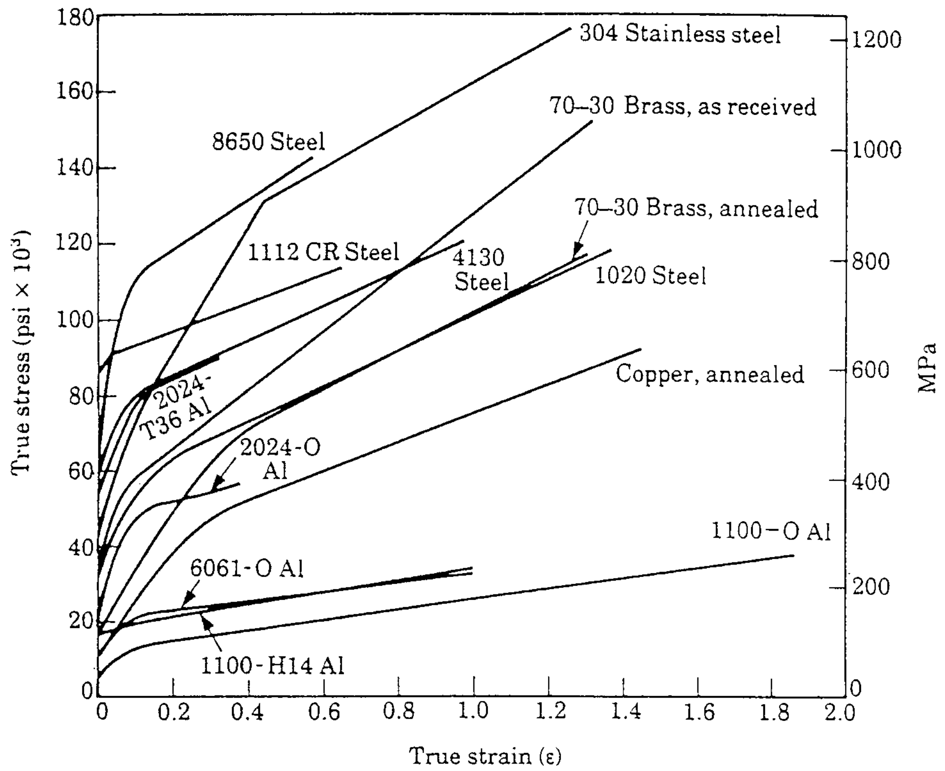

Fig. 3 True stress–true strain curves.

$$\varepsilon = \ln\left(\frac{h_0}{h}\right)$$

$$\text{Roll/Power} = \frac{2\pi FLN}{60{,}000} \quad \text{kW} \qquad (2)$$

where F = newtons
L = meters
N = rev per min

or

$$\text{Power} = \frac{2\pi FLN}{33{,}000} \quad \text{hp} \qquad (3)$$

where F = lb
L = ft

2.3 Forging

Forging is the plastic working of metal by means of localized compressive forces exerted by manual or power hammers, presses, or special forging machines.

Various types of forging have been developed to provide great flexibility, making it economically possible to forge a single piece or to mass produce thousands of identical parts. The metal may be

1. Drawn out, increasing its length and decreasing its cross section
2. Upset, increasing the cross section and decreasing the length, or
3. Squeezed in closed impression dies to produce multidirectional flow

The state of stress in the work is primarily uniaxial or multiaxial compression. The common forging processes are

1. Open-die hammer
2. Impression-die drop forging
3. Press forging
4. Upset forging
5. Roll forging
6. Swaging

Open-Die Hammer Forging

Open-die forging, (Fig. 4) does not confine the flow of metal, the hammer and anvil often being completely flat. The desired shape is obtained by manipulating the workpiece between blows. Specially shaped tools or a slightly shaped die between the workpiece and the hammer or anvil are used to aid in shaping sections (round, concave, or convex), making holes, or performing cutoff operations.

The force F required for an open-die forging operation on a solid cylindrical piece can be calculated by

$$F = Y_f \pi r^2 \left(1 + \frac{2\mu r}{3h}\right) \tag{4}$$

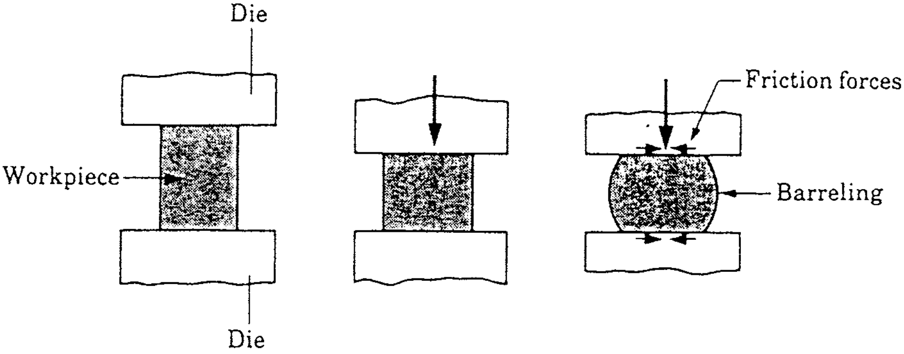

Fig. 4 Open-die hammer forging.

where Y_f = flow stress at the specific ε [$\varepsilon = \ln(h_0/h)$]
μ = coefficient of friction
r and h = radius and height of workpiece

Impression-Die Drop Forging

In impression-die or closed-die drop forging (Fig. 5), the heated metal is placed in the lower cavity of the die and struck one or more blows with the upper die. This hammering causes the metal to flow so as to fill the die cavity. Excess metal is squeezed out between the die faces along the periphery of the cavity to form a flash. When forging is completed, the flash is trimmed off by means of a trimming die.

The forging force F required for impression-die forging can be estimated by

$$F = KY_fA \tag{5}$$

where K = multiplying factor (4–12) depending on the complexity of the shape
Y_f = flow stress at forging temperature
A = projected area, including flash

Press Forging

Press forging employs a slow-squeezing action that penetrates throughout the metal and produces a uniform metal flow. In hammer or impact forging, metal flow is a response to the energy in the hammer–workpiece collision. If all the energy can be dissipated through flow of the surface layers of metal and absorption by the press foundation, the interior regions of the workpiece can go undeformed. Therefore, when the forging of large sections is required, press forging must be employed.

Upset Forging

Upset forging involves increasing the diameter of the end or central portion of a bar of metal by compressing its length. Upset-forging machines are used to

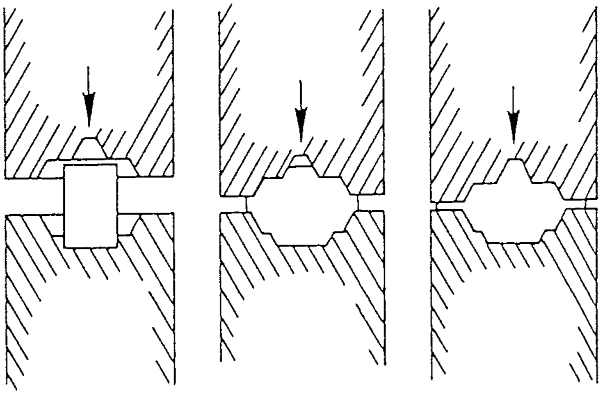

Fig. 5 Impression-die drop forging.

forge heads on bolts and other fasteners, valves, couplings, and many other small components.

Roll Forging

Roll forging, in which round or flat bar stock is reduced in thickness and increased in length, is used to produce such components as axles, tapered levers, and leaf springs.

Swaging

Swaging involves hammering or forcing a tube or rod into a confining die to reduce its diameter, the die often playing the role of the hammer. Repeated blows cause the metal to flow inward and take the internal form of the die.

2.4 Extrusion

In the extrusion process (Fig. 6), metal is compressively forced to flow through a suitably shaped die to form a product with reduced cross section. Although it may be performed either hot or cold, hot extrusion is employed for many metals to reduce the forces required, to eliminate cold-working effects, and to reduce directional properties. The stress state within the material is triaxial compression.

Lead, copper, aluminum, and magnesium, and alloys of these metals, are commonly extruded, taking advantage of the relatively low yield strengths and extrusion temperatures. Steel is more difficult to extrude. Yield strengths are high and the metal has a tendency to weld to the walls of the die and confining chamber under the conditions of high temperature and pressures. With the development and use of phosphate-based and molten glass lubricants, substantial quantities of hot steel extrusions are now produced. These lubricants adhere to the billet and prevent metal-to-metal contact throughout the process.

Almost any cross-section shape can be extruded from the nonferrous metals. Hollow shapes can be extruded by several methods. For tubular products, the stationary or moving mandrel process is often employed. For more complex internal cavities, a spider mandrel or torpedo die is used. Obviously, the cost for hollow extrusions is considerably greater than for solid ones, but a wide variety of shapes can be produced that cannot be made by any other process.

The extrusion force F can be estimated from the formula

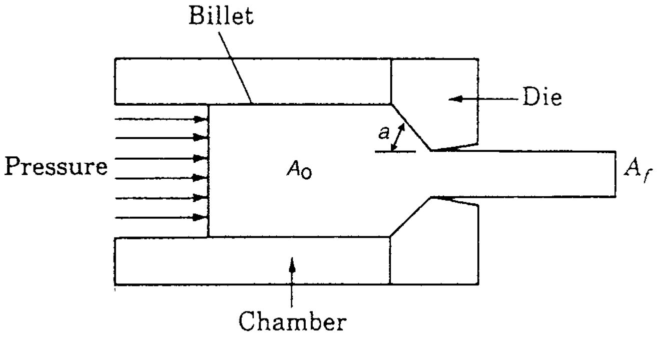

Fig. 6 Extrusion process.

$$F = A_0 k \ln\left(\frac{A_0}{A}\right) \tag{6}$$

where k = extrusion constant depends on material and temperature (see Fig. 7)
A_0 = billet area
A_f = finished extruded area

2.5 Drawing

Drawing (Fig. 8) is a process for forming sheet metal between an edge-opposing punch and a die (draw ring) to produce a cup, cone, box, or shell-like part. The work metal is bent over and wrapped around the punch nose. At the same time, the outer portions of the blank move rapidly toward the center of the blank until they flow over the die radius as the blank is drawn into the die cavity by the punch. The radial movement of the metal increases the blank thickness as the metal moves toward the die radius; as the metal flows over the die radius, this thickness decreases because of the tension in the shell wall between the punch nose and the die radius and (in some instances) because of the clearance between the punch and the die.

The force (load) required for drawing a round cup is expressed by the following empirical equation:

$$L = \pi dtS\left(\frac{D}{d} - k\right) \tag{7}$$

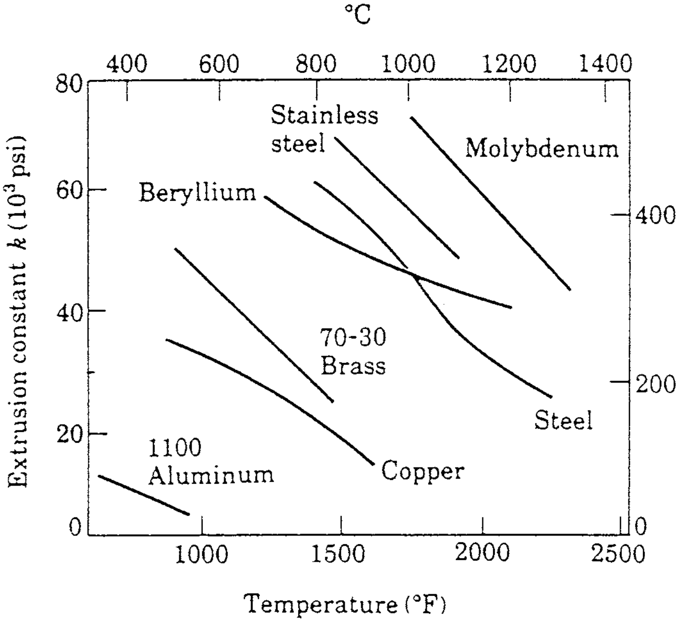

Fig. 7 Extrusion constant k.

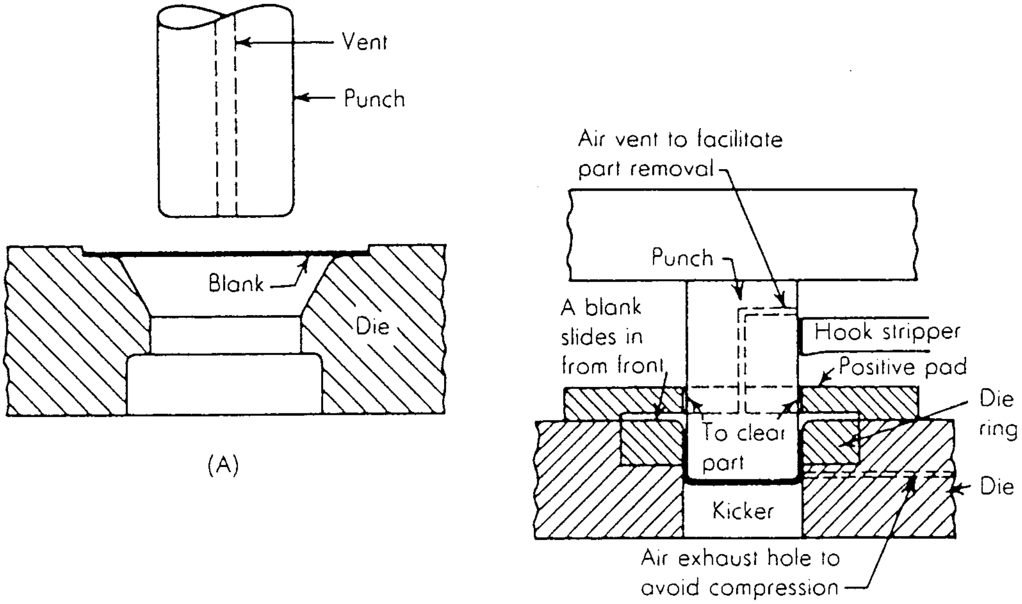

Fig. 8 Drawing process.

where L = press load, lb
d = cup diameter, in.
D = blank diameter, in.
t = work-metal thickness, in.
S = tensile strength, lb/in.^{2}
k = constant that takes into account frictional and bending forces, usually 0.6–0.7

The force (load) required for drawing a rectangular cup can be calculated from the following equation:

$$L = tS(2\pi R k_A + l k_B) \tag{8}$$

where L = press load, lb
t = work-metal thickness, in.
S = tensile strength, lb/in.^{2}
R = corner radius of the cup, in.
l = the sum of the lengths of straight sections of the sides, in.
k_A and k_B = constants

Values for k_A range from 0.5 (for a shallow cup) to 2.0 (for a cup of depth five to six times the corner radius). Values for k_B range from 0.2 (for easy draw radius, ample clearance, and no blank-holding force) and 0.3 (for similar free flow and normal blank-holding force of about $L/3$) to a maximum of 1.0 (for metal clamped too tightly to flow).

Figure 9 can be used as a general guide for computing maximum drawing load for a round shell. These relations are based on a free draw with sufficient clearance so that there is no ironing, using a maximum reduction of 50%. The nomograph gives the load required to fracture the cup (1 ton = 8.9 kN).

Blank Diameters

The following equations may be used to calculate the blank size for cylindrical shells of relatively thin metal. The ratio of the shell diameter to the corner radius (d/r) can affect the blank diameter and should be taken into consideration. When d/r is 20 or more,

$$D = \sqrt{d^2 + 4dh} \tag{9}$$

When d/r is between 15 and 20,

$$D = \sqrt{d^2 + 4dh - 0.5r} \tag{10}$$

When d/r is between 10 and 15,

$$D = \sqrt{d^2 + 4dh - r} \tag{11}$$

When d/r is below 10,

$$D = \sqrt{(d - 2r)^2 + 4d(h - r) + 2\pi r(d - 0.7r)} \tag{12}$$

where D = blank diameter
d = shell diameter
h = shell height
r = corner radius

The above equations are based on the assumption that the surface area of the blank is equal to the surface area of the finished shell.

In cases where the shell wall is to be ironed thinner than the shell bottom, the volume of metal in the blank must equal the volume of the metal in the finished shell. Where the wall-thickness reduction is considerable, as in brass shell cases, the final blank size is developed by trial. A tentative blank size for an ironed shell can be obtained from the equation

$$D = \sqrt{d^2 + 4dh\,\frac{t}{T}} \tag{13}$$

where t = wall thickness
T = bottom thickness

2.6 Spinning

Spinning is a method of forming sheet metal or tubing into seamless hollow cylinders, cones, hemispheres, or other circular shapes by a combination of ro-

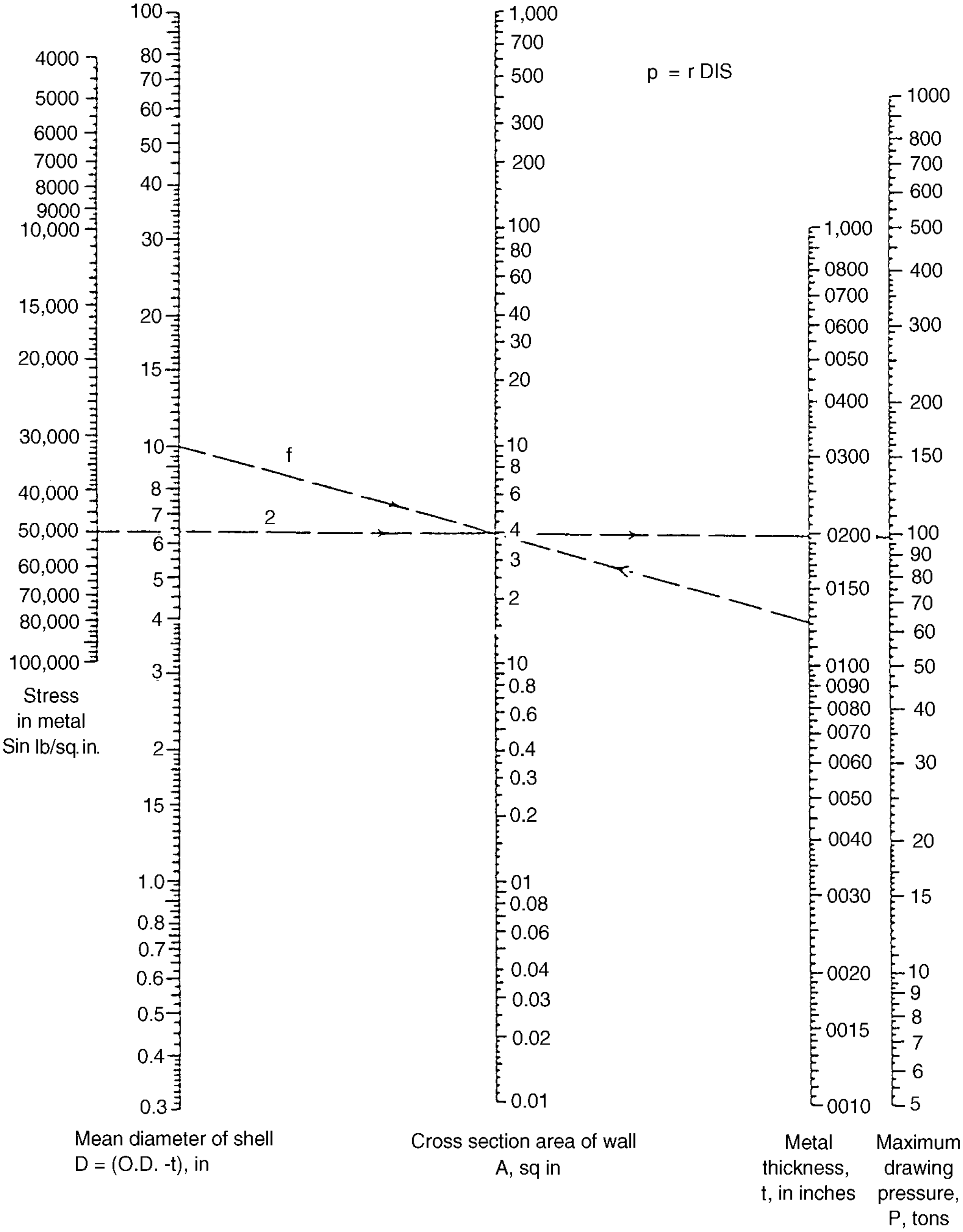

Fig. 9 Nomograph for estimating drawing pressures.

tation and force. On the basis of techniques used, applications, and results obtainable, the method may be divided into two categories: *manual spinning* (with or without mechanical assistance to increase the force) and *power spinning*.

Manual spinning entails no appreciable thinning of metal. The operation ordinarily done in a lathe consists of pressing a tool against a circular metal blank that is rotated by the headstock.

Power spinning is also known as *shear spinning* because in this method metal is intentionally thinned, by shear forces. In power spinning, forces as great as 400 tons are used.

The application of shear spinning to conical shapes is shown schematically in Fig. 10. The metal deformation is such that forming is in accordance with the sine law, which states that the wall thickness of the starting blank and that of the finished workpiece are related as

$$t_2 = t_1(\sin \alpha) \tag{14}$$

where t_1 = thickness of the starting blank
t_2 = thickness of the spun workpiece
α = one-half the apex angle of the cone

Tube Spinning

Tube spinning is a rotary-point method of extruding metal, much like cone spinning, except that the sine law does not apply. Because the half-angle of a cylinder is zero, tube spinning follows a purely volumetric rule, depending on the practical limits of deformation that the metal can stand without intermediate annealing.

2.7 Pipe Welding

Large quantities of small-diameter steel pipe are produced by two processes that involve hot forming of metal strip and welding of its edges through utilization

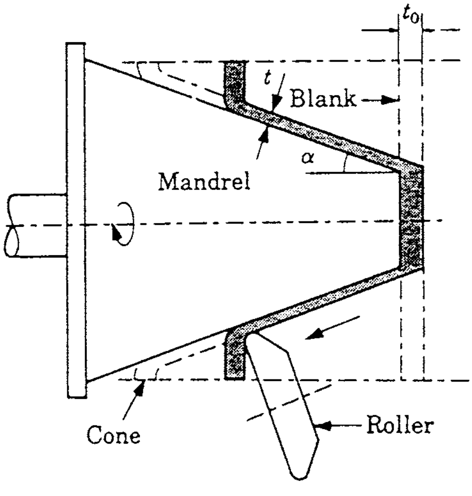

Fig. 10 Setup and dimensional relations for one-operation power spinning of a cone.

of the heat contained in the metal. Both of these processes, *butt welding* and *lap welding* of pipe, utilize steel in the form of skelp—long and narrow strips of the desired thickness. Because the skelp has been previously hot rolled and the welding process produces further compressive working and recrystallization, pipe welding by these processes is uniform in quality.

In the butt-welded pipe process, the skelp is unwound from a continuous coil and is heated to forging temperatures as it passes through a furnace. Upon leaving the furnace, it is pulled through forming rolls that shape it into a cylinder. The pressure exerted between the edges of the skelp as it passes through the rolls is sufficient to upset the metal and weld the edges together. Additional sets of rollers size and shape the pipe. Normal pipe diameters range from $\frac{1}{8}$–3 in. (3–75 mm).

The lap-welding process for making pipe differs from butt welding in that the skelp has beveled edges and a mandrel is used in conjunction with a set of rollers to make the weld. The process is used primarily for larger sizes of pipe, from about 2–14 in. (50–400 mm) in diameter.

2.8 Piercing

Thick-walled and seamless tubing is made by the piercing process. A heated, round billet, with its leading end center-punched, is pushed longitudinally between two large, convex-tapered rolls that revolve in the same direction, their axes being inclined at opposite angles of about 6° from the axis of the billet. The clearance between the rolls is somewhat less than the diameter of the billet. As the billet is caught by the rolls and rotated, their inclination causes the billet to be drawn forward into them. The reduced clearance between the rolls forces the rotating billet to deform into an elliptical shape. To rotate with an elliptical cross section, the metal must undergo shear about the major axis, which causes a crack to open. As the crack opens, the billet is forced over a pointed mandrel that enlarges and shapes the opening, forming a seamless tube (Fig. 11).

This procedure applies to seamless tubes up to 6 in. (150 mm) in diameter. Larger tubes up to 14 in. (355 mm) in diameter are given a second operation on piercing rolls. To produce sizes up to 24 in. (610 mm) in diameter, reheated, double-pierced tubes are processed on a rotary rolling mill, and are finally completed by reelers and sizing rolls, as described in the single-piercing process.

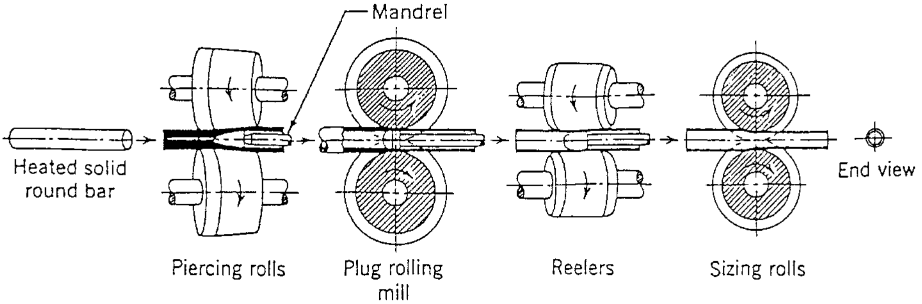

Fig. 11 Principal steps in the manufacture of seamless tubing.

3 COLD-WORKING PROCESSES

Cold working is the plastic deformation of metals below the recrystallization temperature. In most cases of manufacturing, such cold forming is done at room temperature. In some cases, however, the working may be done at elevated temperatures that will provide increased ductility and reduced strength, but will be below the recrystallization temperature.

When compared to hot working, cold-working processes have certain distinct advantages:

1. No heating required
2. Better surface finish obtained
3. Superior dimension control
4. Better reproducibility and interchangeability of parts
5. Improved strength properties
6. Directional properties can be imparted
7. Contamination problems minimized

Some disadvantages associated with cold-working processes include:

1. Higher forces required for deformation
2. Heavier and more powerful equipment required
3. Less ductility available
4. Metal surfaces must be clean and scale-free
5. Strain hardening occurs (may require intermediate anneals)
6. Imparted directional properties may be detrimental
7. May produce undesirable residual stresses

3.1 Classification of Cold-Working Operations

The major cold-working operations can be classified basically under the headings of squeezing, bending, shearing, and drawing, as follows:

Squeezing	*Bending*	*Shearing*	*Drawing*
Rolling	Angle	Shearing Slitting	Bar and tube drawing
Swaging	Roll	Blanking	Wire drawing
Cold forging	Roll forming	Piercing Lancing Perforating	Spinning
Sizing	Drawing	Notching Nibbling	Embossing
Extrusion	Seaming	Shaving	Stretch forming
Riveting	Flanging	Trimming	Shell drawing
Staking	Straightening	Cutoff	Ironing
Coining		Dinking	High-energy rate forming
Peening			
Burnishing			
Die hobbing			
Thread rolling			

3.2 Squeezing Processes

Most of the cold-working squeezing processes have identical hot-working counterparts or are extensions of them. The primary reasons for deforming cold rather than hot are to obtain better dimensional accuracy and surface finish. In many cases, the equipment is basically the same, except that it must be more powerful.

Cold Rolling

Cold rolling accounts for by far the greatest tonnage of cold-worked products. Sheets, strip, bars, and rods are cold rolled to obtain products that have smooth surfaces and accurate dimensions.

Swaging

Swaging basically is a process for reducing the diameter, tapering, or pointing round bars or tubes by external hammering. A useful extension of the process involves the formation of internal cavities. A shaped mandrel is inserted inside a tube and the tube is then collapsed around it by swaging (Fig. 12).

Cold Forging

Extremely large quantities of products are made by cold forging, in which the metal is squeezed into a die cavity that imparts the desired shape. Cold heading is used for making enlarged sections on the ends of rod or wire, such as the heads on bolts, nails, rivets, and other fasteners.

Sizing

Sizing involves squeezing areas of forgings or ductile castings to a desired thickness. It is used principally on basses and flats, with only enough deformation to bring the region to a desired dimension.

Extrusion

This process is often called *impact extrusion* and was first used only with the low-strength ductile metals, such as lead, tin, and aluminum, for producing such items as collapsible tubes for toothpaste, medications, and so forth; small "cans" such as are used for shielding in electronics and electrical apparatus; and larger cans for food and beverages. In recent years, cold extrusion has been used for forming mild steel parts, often being combined with cold heading.

Another type of cold extrusion, known as *hydrostatic extrusion,* used high fluid pressure to extrude a billet through a die, either into atmospheric pressure

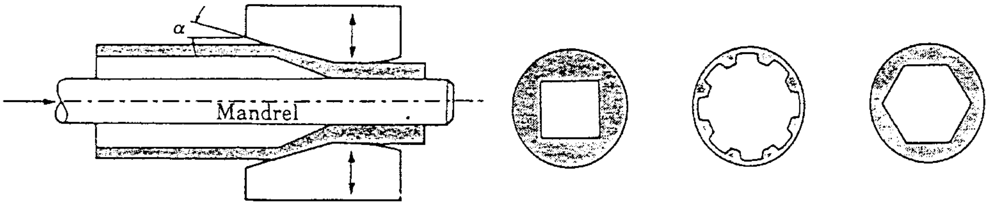

Fig. 12 Cross sections of tubes produced by swaging on shaped mandrels. Rifling (spiral grooves) in small gun barrels can be made by this process.

or into a lower-pressure chamber. The pressure-to-pressure process makes possible the extrusion of relatively brittle materials, such as molybdenum, beryllium, and tungsten. Billet-chamber friction is eliminated, billet-die lubrication is enhanced by the pressure, and the surrounding pressurized atmosphere suppresses crack initiation and growth.

Riveting

In riveting, a head is formed on the shank end of a fastener to provide a permanent method of joining sheets or plates of metal together. Although riveting usually is done hot in structural work, in manufacturing it almost always is done cold.

Staking

Staking is a commonly used cold-working method for permanently fastening two parts together where one protrudes through a hole in the other. A shaped punch is driven into one of the pieces, deforming the metal sufficiently to squeeze it outward.

Coining

Coining involves cold working by means of positive displacement punch while the metal is completely confined within a set of dies.

Peening

Peening involves striking the surface repeated blows by impelled shot or a round-nose tool. The highly localized blows deform and tend to stretch the metal surface. Because the surface deformation is resisted by the metal underneath, the result is a surface layer under residual compression. This condition is highly favorable to resist cracking under fatigue conditions, such as repeated bending, because the compressive stresses are subtractive from the applied tensile loads. For this reason, shafting, crankshafts, gear teeth, and other cyclic-loaded components are frequently peened.

Burnishing

Burnishing involves rubbing a smooth, hard object under considerable pressure over the minute surface protrusions that are formed on a metal surface during machining or shearing, thereby reducing their depth and sharpness through plastic flow.

Hobbing

Hobbing is a cold-working process that is used to form cavities in various types of dies, such as those used for molding plastics. A male hob is made with the contour of the part that ultimately will be formed by the die. After the hob is hardened, it is slowly pressed into an annealed die block by means of hydraulic press until the desired impression is produced.

Thread Rolling

Threads can be rolled in any material sufficiently plastic to withstand the forces of cold working without disintegration. Threads can be rolled by flat or roller dies.

3.3 Bending

Bending is the uniform straining of material, usually flat sheet or strip metal, around a straight axis that lies in the neutral plane and normal to the lengthwise direction of the sheet or strip. Metal flow takes place within the plastic range of the metal, so that the bend retains a permanent set after removal of the applied stress. The inner surface of the bend is in compression; the outer surface is in tension.

Terms used in bending are defined and illustrated in Fig. 13. The neutral axis is the plane area in bent metal where all strains are zero.

Bend Allowances

Since bent metal is longer after bending, its increased length, generally of concern to the product designer, may also have to be considered by the die designer if the length tolerance of the bent part is critical. The length of bent metal may be calculated from the equation

$$B = \frac{A}{360} \times 2\pi(R_i + Kt) \tag{15}$$

where B = bend allowance, in. (mm) (along neutral axis)
A = bend angle, deg
R_i = inside radius of bend, in. (mm)
t = metal thickness, in. (mm)
K = 0.33 when R_i is less than $2t$ and is 0.50 when R_i is more than $2t$

Bending Methods

Two bending methods are commonly made use of in press tools. Metal sheet or strip, supported by a V block (Fig. 14), is forced by a wedge-shaped punch into the block.

Edge bending (Fig. 14) is cantilever loading of a beam. The bending punch (1) forces the metal against the supporting die (2).

Bending Force

The force required for V bending is as follows:

$$P = \frac{KLSt^2}{W} \tag{16}$$

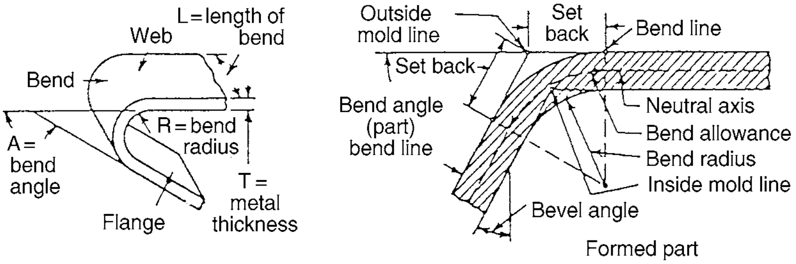

Fig. 13 Bend terms.

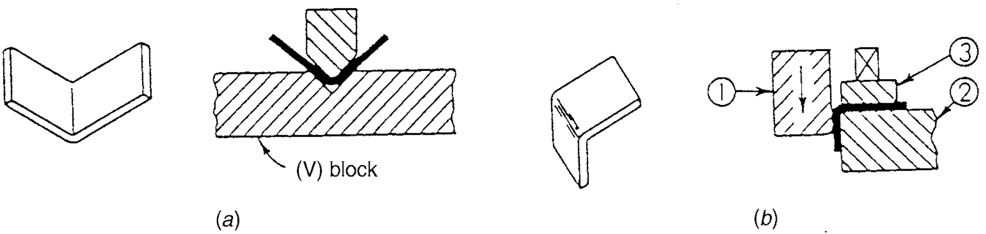

Fig. 14 Bending methods: (*a*) V bending and (*b*) edge bending.

where P = bending force, tons (for metric usage, multiply number of tons by 8.896 to obtain kilonewtons)
K = die opening factor: 1.20 for a die opening of 16 times metal thickness, 1.33 for an opening of eight times metal thickness
L = length of part, in.
S = ultimate tensile strength, tons/in.^{2}
W = width of V or U die, in.
t = metal thickness, in.

For U bending (channel bending), pressures will be approximately twice those required. For U bending, edge bending is required about one-half those needed for V bending. Table 1 gives the ultimate strength = S for various materials.

Several factors must be considered when designing parts that are to be made by bending. Of primary importance is the minimum radius that can be bent successfully without metal cracking. This, of course, is related to the ductility of the metal.

Angle Bending

Angle bends up to 150° in the sheet metal under about $\frac{1}{16}$ in. (1.5 mm) in thickness may be made in a bar folder. Heavier sheet metal and more complex bends in thinner sheets are made on a press brake.

Roll Bending

Plates, heavy sheets, and rolled shapes can be bent to a desired curvature on forming rolls. These usually have three rolls in the form of a pyramid, with the two lower rolls being driven and the upper roll adjustable to control the degree

Table 1 **Ultimate Strength**

Metal	(ton/in.^{2})
Aluminum and alloys	6.5–38.0
Brass	19.0–38.0
Bronze	31.5–47.0
Copper	16.0–25.0
Steel	22.0–40.0
Tin	1.1–1.4
Zinc	9.7–13.5

of curvature. Supports can be swung clear to permit removal of a closed shape from the rolls. Bending rolls are available in a wide range of sizes, some being capable of bending plate up to 6 in. (150 mm) thick.

Cold-Roll Forming

This process involves the progressive bending of metal strip as it passes through a series of forming rolls. A wide variety of moldings, channeling, and other shapes can be formed on machines that produce up to 10,000 ft (3000 m) of product per day.

Seaming

Seaming is used to join ends of sheet metal to form containers such as cans, pails, and drums. The seams are formed by a series of small rollers on seaming machines that range from small hand-operated types to large automatic units capable of producing hundreds of seams per minute in the mass production of cans.

Flanging

Flanges can be rolled on sheet metal in essentially the same manner as seaming is done. In many cases, however, the forming of flanges and seams involves drawing, since localized bending occurs on a curved axis.

Straightening

Straightening or flattening has as its objective the opposite of bending and often is done before other cold-forming operations to ensure that flat or straight material is available. Two different techniques are quite common. *Roll straightening* or *roller leveling* involves a series of reverse bends. The rod, sheet, or wire is passed through a series of rolls having decreased offsets from a straight line. These bend the metal back and forth in all directions, stressing it slightly beyond its previous elastic limit and thereby removing all previous permanent set.

Sheet may also be straightened by a process called *stretcher leveling.* The sheets are grabbed mechanically at each end and stretched slightly beyond the elastic limit to remove previous stresses and thus produce the desired flatness.

3.4 Shearing

Shearing is the mechanical cutting of materials in sheet or plate form without the formation of chips or use of burning or melting. When the two cutting blades are straight, the process is called *shearing.* Other processes, in which the shearing blades are in the form of curved edges or punches and dies, are called by other names, such as *blanking, piercing, notching, shaving,* and *trimming.* These all are basically shearing operations, however.

The required shear force can be calculated as

$$F = \left(\frac{S \times P \times t^2 \times 12}{R}\right)\left(1 - \frac{P}{2}\right) \tag{17}$$

where F = shear force, lb
S = shear strength (stress), psi

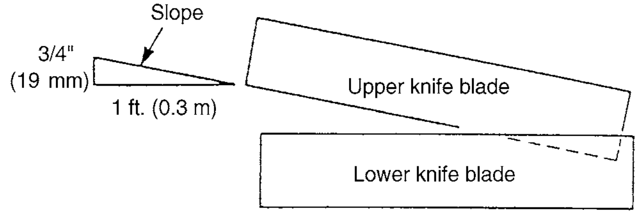

Fig. 15 Rake is the angular slope formed by the cutting edges of the upper and lower knives.

P = penetration of knife into material, %
t = thickness of material, in.
R = rake of the knife blade, in./ft (Fig. 15)

For SI units, the force is multiplied by 4.448 to obtain newtons (N). Table 2 gives the values of P and S for various materials.

Blanking

A blank is a shape cut from flat or preformed stock. Ordinarily, a blank serves as a starting workpiece for a formed part; less often, it is a desired end product.

Table 2 Values of Percent Penetration and Shear Strength for Various Materials

Material	Percent Penetration	Shear Strength, psi (MPa)
Lead alloys	50	3500 (24.1)–6000 (41.3)
Tin alloys	40	5000 (34.5)–10,000 (69)
Aluminum alloys	60	8000 (55.2)–45,000 (310)
Titanium alloys	10	60,000 (413)–70,000 (482)
Zinc	50	14,000 (96.5)
Cold worked	25	19,000 (131)
Magnesium alloys	50	17,000 (117)–30,000 (207)
Copper	55	22,000 (151.7)
Cold worked	30	28,000 (193)
Brass	50	32,000 (220.6)
Cold worked	30	52,000 (358.5)
Tobin bronze	25	36,000 (248.2)
Cold worked	17	42,000 (289.6)
Steel, 0.10C	50	35,000 (241.3)
Cold worked	38	43,000 (296.5)
Steel, 0.40C	27	62,000 (427.5)
Cold worked	17	78,000 (537.8)
Steel, 0.80C	15	97,000 (668.8)
Cold worked	5	127,000 (875.6)
Steel, 1.00C	10	115,000 (792.9)
Cold worked	2	150,000 (1034.2)
Silicon steel	30	65,000 (448.2)
Stainless steel	30	57,000 (363)–128,000 (882)
Nickel	55	35,000 (241.3)

Calculation of the forces and the work involved in blanking gives average figures that are applicable only when (a) the correct shear strength for the material is used, and (b) the die is sharp and the punch is in good condition, has correct clearance, and is functioning properly.

The total load on the press, or the press capacity required to do a particular job, is the sum of the cutting force and other forces acting at the same time, such as the blankholding force exerted by a die cushion.

Cutting Force: Square-End Punches and Dies

When punch and die surfaces are flat and at right angles to the motion of the punch, the cutting force can be found by multiplying the area of the cut section by the shear strength of the work material:

$$L = Stl \tag{18}$$

where L = load on the press, lb (cutting force)
S = shear strength of the stock, psi
t = stock thickness, in.
l = the length or perimeter of cut, in.

Piercing

Piercing is a shearing operation wherein the shearing blades take the form of closed, curved lines on the edges of a punch and die. Piercing is basically the same as blanking except that the piece punched out is the scrap and the remainder of the strip becomes the desired workpiece.

Lancing

Lancing is a piercing operation that may take the form of a slit in the metal or an actual hole. The purpose of lancing is to permit adjacent metal to flow more readily in subsequent forming operations.

Perforating

Perforating consists of piercing a large number of closely spaced holes.

Notching

Notching is essentially the same as piercing except that the edge of the sheet of metal forms a portion of the periphery of the piece that is punched out. It is used to form notches of any desired shape along the edge of a sheet.

Nibbling

Nibbling is a variation of notching in which a special machine makes a series of overlapping notches, each farther into the sheet of metal.

Shaving

Shaving is a finished operation in which a very small amount of metal is sheared away around the edge of a blanked part. Its primary use is to obtain greater dimensional accuracy, but it also may be used to obtain a square of smoother edge.

Trimming

Trimming is used to remove the excess metal that remains after a drawing, forging, or casting operation. It is essentially the same as blanking.

Cutoff

A cutoff operation is one in which a stamping is removed from a strip of stock by means of a punch and die. The cutoff punch and die cut across the entire width of the strip. Frequently, an irregularly shaped cutoff operation may simultaneously give the workpiece all or part of the desired shape.

Dinking

Dinking is a modified shearing operation that is used to blank shapes from low-strength materials, primarily rubber, fiber, and cloth.

3.5 Drawing

Cold Drawing

Cold drawing is a term that can refer to two somewhat different operations. If the stock is in the form of sheet metal, cold drawing is the forming of parts wherein plastic flow occurs over a curved axis. This is one of the most important of all cold-working operations because a wide range of parts, from small caps to large automobile body tops and fenders, can be drawn in a few seconds each. Cold drawing is similar to hot drawing, but the higher deformation forces, thinner metal, limited ductility, and closer dimensional tolerance create some distinctive problems.

If the stock is wire, rod, or tubing, *cold drawing* refers to the process of reducing the cross section of the material by pulling it through a die, a sort of tensile equivalent to extrusion.

Cold Spinning

Cold spinning is similar to hot spinning, discussed above.

Stretch Forming

In stretch forming, only a single male form block is required. The sheet of metal is gripped by two or more sets of jaws that stretch it and wrap it around the form block as the latter raises upward. Various combinations of stretching, wrapping, and upward motion of the blocks are used, depending on the shape of the part.

Shell or Deep Drawing

The drawing of closed cylindrical or rectangular containers, or a variation of these shapes, with a depth frequently greater than the narrower dimension of their opening, is one of the most important and widely used manufacturing processes. Because the process had its earliest uses in manufacturing artillery shells and cartridge cases, it is sometimes called *shell drawing*. When the depth of the drawn part is less than the diameter, or minimum surface dimension, of the blank, the process is considered to be *shallow drawing*. If the depth is greater than the diameter, it is considered to be *deep drawing*.

The design of complex parts that are to be drawn has been aided considerably by computer techniques, but is far from being completely and successfully solved. Consequently, such design still involves a mix of science, experience, empirical data, and actual experimentation. The body of known information is quite substantial, however, and is being used with outstanding results.

Forming with Rubber or Fluid Pressure

Several methods of forming use rubber or fluid pressure (Fig. 16) to obtain the desired information and thereby eliminate either the male or female member of the die set. Blanks of sheet metal are placed on top of form blocks, which usually are made of wood. The upper ram, which contains a pad of rubber 8–10 in. (200–250 mm) thick in a steel container, then descends. The rubber pad is confined and transmits force to the metal, causing it to bend to the desired shape. Since no female die is used and form blocks replace the male die, die cost is quite low.

The hydroform process or "rubber bag forming" replaces the rubber pad with a flexible diaphragm backed by controlled hydraulic pressure. Deeper parts can be formed with truly uniform fluid pressure.

The bulging oil or rubber is used for applying an internal bulging force to expand a metal blank or tube outward against a female mold or die, thereby eliminating the necessity for a complicated, multiple-piece male die member.

Ironing

Ironing is the name given to the process of thinning the walls of a drawn cylinder by passing it between a punch and a die where the separation is less than the original wall thickness. The walls are elongated and thinned while the base remains unchanged. The most common example of an ironed product is the thin-walled all-aluminum beverage can.

Embossing

Embossing is a method for producing lettering or other designs in thin sheet metal. Basically, it is a very shallow drawing operation, usually in open dies, with the depth of the draw being from one to three times the thickness of the metal.

High-Energy-Rate Forming

A number of methods have been developed for forming metals through the release and application of large amounts of energy in a very short interval (Fig.

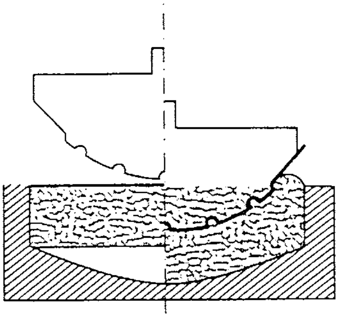

Fig. 16 Form with rubber.

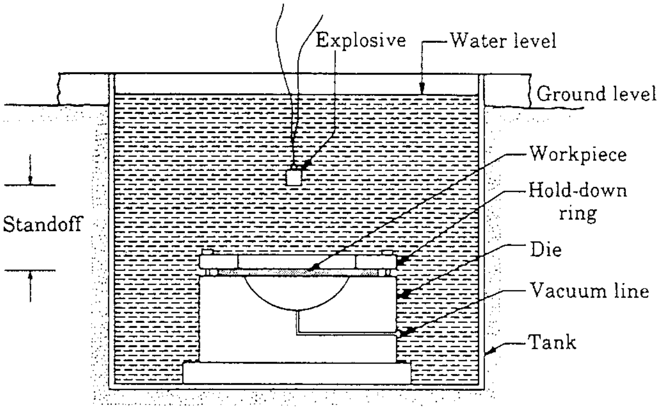

Fig. 17 High-energy-rate forming.

17). These processes are called *high-energy-rate forming processes* (HERF). Many metals tend to deform more readily under the ultrarapid rates of load application used in these processes, a phenomenon apparently related to the relative rates of load application and the movement of dislocations through the metal. As a consequence, HERF makes it possible to form large workpieces and difficult-to-form metals with less expensive equipment and tooling than would otherwise be required.

The high energy-release rates are obtained by five methods:

1. Underwater explosions
2. Underwater spark discharge (electrohydraulic techniques)
3. Pneumatic–mechanical means
4. Internal combustion of gaseous mixtures
5. Rapidly formed magnetic fields (electromagnetic techniques)

4 METAL CASTING AND MOLDING PROCESSES

Casting provides a versatility and flexibility that have maintained casting position as a primary production method for machine elements. Casting processes are divided according to the specific type of molding method used in casting, as follows:

1. Sand
2. Centrifugal
3. Permanent
4. Die
5. Plaster-mold
6. Investment

4.1 Sand Casting

Sand casting consists basically of pouring molten metal into appropriate cavities formed in a sand mold (Fig. 18). The sand may be natural, synthetic, or an artificially blended material.

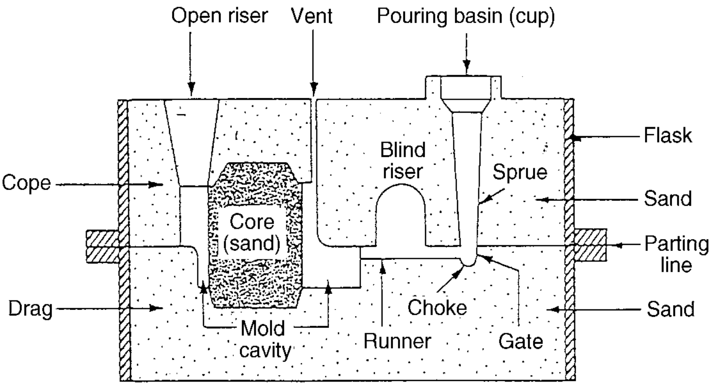

Fig. 18 Sectional view of casting mold.

Molds

The two common types of sand molds are the *dry sand mold* and the *green sand mold.* In the dry sand mold, the mold is dried thoroughly prior to closing and pouring, while the green sand mold is used without any preliminary drying. Because the dry sand mold is more firm and resistant to collapse than the green sand mold, core pieces for molds are usually made in this way. Cores are placed in mold cavities to form the interior surfaces of castings.

Patterns

To produce a mold for a conventional sand-cast part, it is necessary to make a pattern of the part. Patterns are made from wood or metal to suit a particular design, with allowances to compensate for such factors as natural metal shrinkage and contraction characteristics. These and other effects, such as mold resistance, distortion, casting design, and mold design, which are not entirely within the range of accurate prediction, generally make it necessary to adjust the pattern in order to produce castings of the required dimensions.

Access to the mold cavity for entry of the molten metal is provided by sprues, runners, and gates.

Shrinkage

Allowances must be made on patterns to counteract the contraction in size as the metal cools. The amount of shrinkage is dependent on the design of the coating, type of metal used, solidification temperature, and mold resistance. Table 3 gives average shrinkage allowance values used in sand casting. Smaller values apply generally to large or cored castings of intricate design. Larger values apply to small to medium simple castings designed with unrestrained shrinkage.

Machining

Allowances are required in many cases because of unavoidable surface impurities, warpage, and surface variations. Average machining allowances are given in Table 4. Good practice dictates use of minimum section thickness compatible

Table 3 Pattern Shrinkage Allowance (in./ft)

Metal	Shrinkage
Aluminum alloys	$\frac{1}{10}-\frac{5}{32}$
Beryllium copper	$\frac{1}{8}-\frac{5}{32}$
Copper alloys	$\frac{3}{16}-\frac{7}{32}$
Everdur	$\frac{3}{16}$
Gray irons	$\frac{1}{8}$
Hastelloy alloys	$\frac{1}{4}$
Magnesium alloys	$\frac{1}{8}-\frac{11}{64}$
Malleable irons	$\frac{1}{16}-\frac{3}{16}$
Meehanite	$\frac{1}{10}-\frac{5}{32}$
Nickel and nickel alloys	$\frac{1}{4}$
Steel	$\frac{1}{8}-\frac{1}{4}$
White irons	$\frac{3}{16}-\frac{1}{4}$

with the design. The normal minimum section recommended for various metals is shown in Table 5.

4.2 Centrifugal Casting

Centrifugal casting consists of having a sand, metal, or ceramic mold that is rotated at high speeds. When the molten metal is poured into the mold, it is thrown against the mold wall, where it remains until it cools and solidifies. The process is increasingly being used for such products as cast-iron pipes, cylinder liners, gun barrels, pressure vessels, brake drums, gears, and flywheels. The metals used include almost all castable alloys. Most dental tooth caps are made by a combined lost-wax process and centrifugal casting.

Table 4 Machining Allowances for Sand Castings (in.)

Metal	Casting Size (in.)	Finish Allowance
Cast irons	Up to 12	$\frac{3}{32}$
	13–24	$\frac{1}{8}$
	25–42	$\frac{3}{16}$
	43–60	$\frac{1}{4}$
	61–80	$\frac{5}{16}$
	81–120	$\frac{3}{8}$
Cast steels	Up to 12	$\frac{1}{8}$
	13–24	$\frac{3}{16}$
	25–42	$\frac{5}{16}$
	43–60	$\frac{3}{8}$
	61–80	$\frac{7}{16}$
	81–120	$\frac{1}{2}$
Malleable irons	Up to 8	$\frac{1}{16}$
	9–12	$\frac{3}{32}$
	13–24	$\frac{1}{8}$
	25–36	$\frac{3}{16}$
Nonferrous metals	Up to 12	$\frac{1}{16}$
	13–24	$\frac{1}{8}$
	25–36	$\frac{5}{32}$

Table 5 Minimum Sections for Sand Castings (in.)

Metal	Section
Aluminum alloys	$\frac{3}{16}$
Copper alloys	$\frac{3}{32}$
Gray irons	$\frac{1}{8}$
Magnesium alloys	$\frac{5}{32}$
Malleable irons	$\frac{1}{8}$
Steels	$\frac{1}{4}$
White irons	$\frac{1}{8}$

Advantages and Limitations

Because of the relatively fast cooling time, centrifugal castings have a fine grain size. There is a tendency for the lighter nonmetallic inclusion, slag particles, and dross to segregate toward the inner radius of the castings (Fig. 19), where it can be easily removed by machining. Owing to the high purity of the outer skin, centrifugally cast pipes have a high resistance to atmospheric corrosion. Figure 19 shows a schematic sketch of how a pipe would be centrifugally cast in a horizontal mold.

Parts that have diameters exceeding their length are produced by vertical-axis casting (see Fig. 20).

If the centrifugal force is too low or too great, abnormalities will develop. Most horizontal castings are spun so that the force developed is about 65g. Vertically cast parts force is about 90–100g.

The centrifugal force (CF) is calculated from

$$\text{CF} = \frac{mv^2}{r}\ lb$$

$$m = \text{Mass} = \frac{W}{g} = \frac{\text{Weight, lb}}{\text{Acceleration of gravity (ft/s)}^2} = \frac{W}{32.2}$$

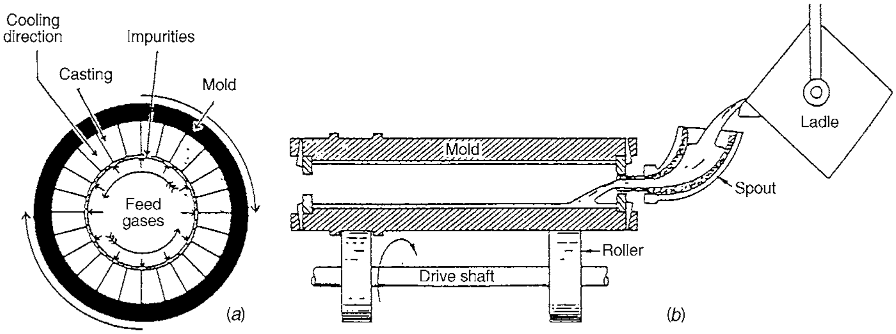

Fig. 19 Principle of centrifugal casting is to produce the high-grade metal by throwing the heavier metal outward and forcing the impurities to congregate inward (*a*). Shown at (*b*) is a schematic of how a horizontal-bond centrifugal casting is made.

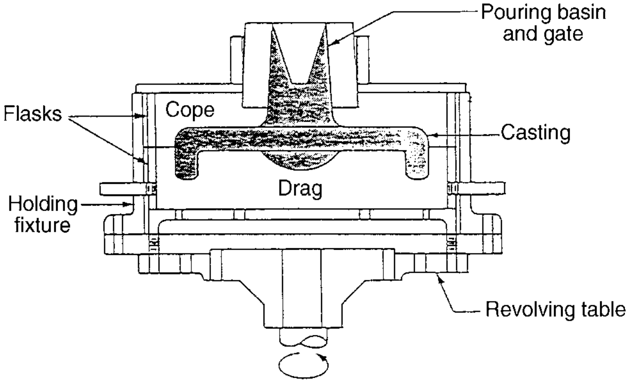

Fig. 20 Floor-type vertical centrifugal casting machine for large-diameter parts.

where v = velocity, ft/s = $r \times w$
r = radius, ft = $\frac{1}{2}$ D
W = angular velocity, rad/s
$W = 2\pi/60 \times$ rpm
D = inside diameter, ft

The number of g is

$$g = \mathrm{CF}/W$$

Hence,

$$\begin{aligned} g &= \frac{1}{W} \times \left[\frac{W}{32.2 \times r}\left(\frac{r \times 2\pi}{60}\right)^2\right] \\ &= r \times 3.41 \times 10^{-4}\ \mathrm{rpm}^2 \\ &= 1.7 \times 10^{-4} \times D \times (\mathrm{rpm})^2 \end{aligned}$$

The spinning speed for horizontal-axis molds may be found in English units from the equation

$$N = \sqrt{(\text{Number of } g) \times \frac{70{,}500}{D}}$$

where N = rpm
D = inside diameter of mold, ft

4.3 Permanent-Mold Casting

As demand for quality castings in production quantities increased, the attractive possibilities of metal molds brought about the development of the permanent-mold process. Although not as flexible regarding design as sand casting, metal-mold casting made possible the continuous production of quantities of casting from a single mold as compared to batch production of individual sand molds.

Metal Molds and Cores

In permanent-mold casting, both metal molds and cores are used, the metal being poured into the mold cavity with the usual gravity head as in sand casting. Molds are normally made of dense iron or meehanite, large cores of cast iron, and small or collapsible cores of alloy steel. All necessary sprues, runners, gates, and risers must be machined into the mold, and the mold cavity itself is made with the usual metal-shrinkage allowances. The mold is usually composed of one, two, or more parts, which may swing or slide for rapid operation. Whereas in sand casting the longest dimension is always placed in a horizontal position, in permanent-mold casting the longest dimension of a part is normally placed in a vertical position.

Production Quantities

Wherever quantities are in the range of 500 pieces or more, permanent-mold casting becomes competitive in cost with sand casting, and if the design is simple, runs as small as 200 pieces are often economical. Production runs of 1000 pieces or more will generally produce a favorable cost difference. High rates of production are possible, and multiple-cavity dies with as many as 16 cavities can be used. In casting gray iron in multiple molds, as many as 50,000 castings per cavity are common with small parts. With larger parts of gray iron, weighing from 12 to 15 lb, single-cavity molds normally yield 2000–3000 pieces per mold on an average. Up to 100,000 parts per cavity or more are not uncommon with nonferrous metals, magnesium providing the longest die life. Low-pressure permanent-mold casting is economical for quantities up to 40,000 pieces (Fig. 21).

Die Casting

Die casting may be classified as a permanent-mold casting system; however, it differs from the process just described in that the molten metal is forced into

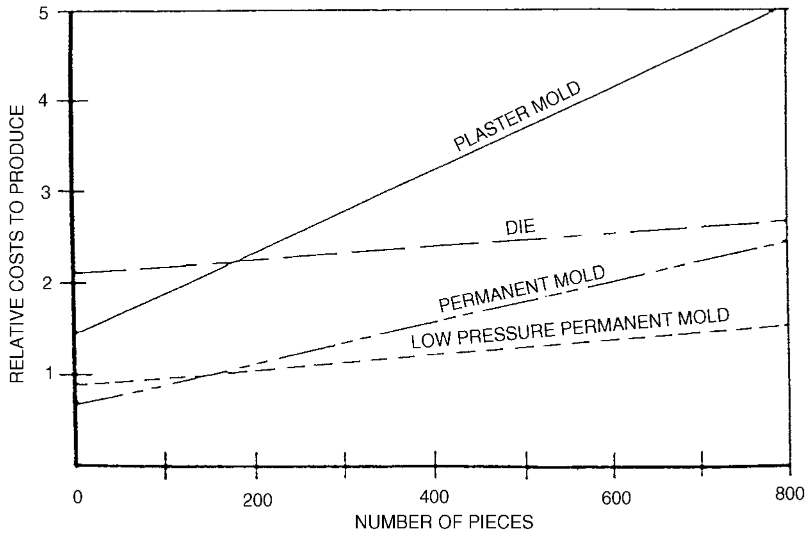

Fig. 21 Cost comparison of various casting systems.

the mold or die under high pressure [1000–30,000 psi (6.89–206.8 MPa)]. The metal solidifies rapidly (within a fraction of a second) because the die is water-cooled. Upon solidification, the die is opened and ejector pins automatically knock the casting out of the die. If the parts are small, several of them may be made at one time in what is termed a *multicavity die.*

There are two main types of machines used: the hot-chamber and the cold-chamber types.

Hot-Chamber Die Casting. In the hot-chamber machine, the metal is kept in a heated holding pot. As the plunger descends, the required amount of alloy is automatically forced into the die. As the piston retracts, the cylinder is again filled with the right amount of molten metal. Metals such as aluminum, magnesium, and copper tend to alloy with the steel plunger and cannot be used in the hot chamber.

Cold-Chamber Die Casting. This process gets its name from the fact that the metal is ladled into the cold chamber for each shot. This procedure is necessary to keep the molten-metal contact time with the steel cylinder to a minimum. Iron pickup is prevented, as is freezing of the plunger in the cylinder.

Advantages and Limitations

Die-casting machines can produce large quantities of parts with close tolerances and smooth surfaces. The size is limited only by the capacity of the machine. Most die castings are limited to about 75 lb (34 kg) of zinc; 65 lb (30 kg) of aluminum; and 44 lb (20 kg) of magnesium. Die castings can provide thinner sections than any other casting process. Wall thickness as thin as 0.015 in. (0.38 mm) can be achieved with aluminum in small items. However, a more common range on larger sizes will be 0.105–0.180 in. (2.67–4.57 mm).

Some difficulty is experienced in getting sound castings in the larger capacities. Gases tend to be entrapped, which results in low strength and annoying leaks. Of course, one way to reduce metal sections without sacrificing strength is to design in ribs and bosses. Another approach to the porosity problem has been to operate the machine under vacuum. This process is now being developed.

The surface quality is dependent on that of the mold. Parts made from new or repolished dies may have a surface roughness of 24 μin. (0.61 μm). The high surface finish available means that, in most cases, coatings such as chromeplating, anodizing, and painting may be applied directly. More recently, decorative finishes of texture, as obtained by photoetching, have been applied. The technique has been used to simulate woodgrain finishes, as well as textile and leather finishes, and to obtain checkering and crosshatching.

4.4 Plaster-Mold Casting

In general, the various methods of plaster-mold casting are similar. The plaster, also known as *gypsum* or *calcium sulfate,* is mixed dry with other elements, such as talc, sand, asbestos, and sodium silicate. To this mix is added a controlled amount of water to provide the desired permeability in the mold. The slurry that results is heated and delivered through a hose to the flasks, all surfaces of which have been sprayed with a parting compound. The plaster slurry readily fills in and around the most minute details in the highly polished brass patterns. Following filling, the molds are subjected to a short period of vibration and the slurry sets in 5–10 min.

Molds

Molds are extracted from the flask with a vacuum head, following which drying is completed in a continuous oven. Copes and drags are then assembled, with cores when required, and the castings are poured. Upon solidification, the plaster is broken away and any cores used are washed out with a high-pressure jet of water.

4.5 Investment Casting

Casting processes in which the pattern is used only once are variously referred to as *lost-wax* or *precision-casting* processes. They involve making a pattern of the desired form out of wax or plastic (usually polystyrene). The expendable pattern may be made by pressing the wax into a split mold or by the use of an injection-molding machine. The patterns may be gated together so that several parts can be made at once. A metal flask is placed around the assembled patterns and a refractory mold slurry is poured in to support the patterns and form the cavities. A vibrating table equipped with a vacuum pump is used to eliminate all the air from the mold. Formerly, the standard procedure was to dip the patterns in the slurry several times until a coat was built up. This is called the *investment process.* After the mold material has set and dried, the pattern material is melted and allowed to run out of the mold.

The completed flasks are heated slowly to dry the mold and to melt out the wax, plastic, or whatever pattern material was used. When the molds have reached a temperature of 100°F (37.8°C), they are ready for pouring. Vacuum may be applied to the flasks to ensure complete filling of the mold cavities.

When the metal has cooled, the investment material is removed by vibrating hammers or by tumbling. As with other castings, the gates and risers are cut off and ground down.

Ceramic Process

The ceramic process is somewhat similar to the investment casting in that a creamy ceramic slurry is poured over a pattern. In this case, however, the pattern, made out of plastic, plaster, wood, metal, or rubber, is reusable. The slurry hardens on the pattern almost immediately and becomes a strong green ceramic of the consistency of vulcanized rubber. It is lifted off the pattern while it is still in the rubberlike phase. The mold is ignited with a torch to burn off the volatile portion of the mix. It is then put in a furnace and baked at 1800°F (982°C), resulting in a rigid refractory mold. The mold can be poured while still hot.

Full-Mold Casting

Full-mold casting may be considered a cross between conventional sand casting and the investment technique of using lost wax. In this case, instead of a conventional pattern of wood, metals, or plaster, a polystyrene foam or styrofoam is used. The pattern is left in the mold and is vaporized by the molten metal as it rises in the mold during pouring. Before molding, the pattern is usually coated with a zirconite wash in an alcohol vehicle. The wash produces a relatively tough skin separating the metal from the sand during pouring and cooling. Conventional foundry sand is used in backing up the mold.

5 PLASTIC-MOLDING PROCESSES

Plastic molding is similar in many ways to metal molding. For most molding operations, plastics are heated to a liquid or a semifluid state and are formed in a mold under pressure. Some of the most common molding processes are discussed below.

5.1 Injection Molding

The largest quantity of plastic parts is made by injection molding. Plastic compound is fed in powdered or granular form from a hopper through metering and melting stages and then injected into a mold. After a brief cooling period, the mold is opened and the solidified part is ejected.

5.2 Coinjection Molding

Coinjection molding makes it possible to mold articles with a solid skin of one thermoplastic and a core of another thermoplastic. The skin material is usually solid while the core material contains blowing agents.

The basic process may be one-, two-, or three-channel technology. In one-channel technology, the two melts are injected into the mold, one after the other. The skin material cools and adheres to the colder surface; a dense skin is formed under proper parameter settings. The thickness of the skin can be controlled by adjustment of injection speed, stock temperature, mold temperature, and flow compatibility of the two melts.

In two- and three-channel techniques, both plastic melts may be introduced simultaneously. This allows for better control of wall thickness of the skin, especially in gate areas on both sides of the part.

Injection-Molded Carbon-Fiber Composites

By mixing carbon or glass fibers in injection-molded plastic parts, they can be made lightweight yet stiffer than steel.

5.3 Rotomolding

In rotational molding, the product is formed inside a closed mold that is rotated about two axes as heat is applied. Liquid or powdered thermoplastic or thermosetting plastic is poured into the mold, either manually or automatically.

5.4 Expandable-Bead Molding

The expandable-bead process consists of placing small beads of polystyrene along with a small amount of blowing agent in a tumbling container. The polystyrene beads soften under heat, which allows a blowing agent to expand them. When the beads reach a given size, depending on the density required, they are quickly cooled. This solidifies the polystyrene in its larger foamed size. The expanded beads are then placed in a mold until it is completely filled. The entrance port is then closed and steam is injected, resoftening the beads and fusing them together. After cooling, the finished, expanded part is removed from the mold.

5.5 Extruding

Plastic extrusion is similar to metal extrusion in that a hot material (plastic melt) is forced through a die having an opening shaped to produce a desired cross section. Depending on the material used, the barrel is heated anywhere from 250 to 600°F (121 to 316°C) to transform the thermoplastic from a solid to a melt. At the end of the extruder barrel is a screen pack for filtering and building back pressure. A breaker plate serves to hold the screen pack in place and straighten the helical flow as it comes off the screen.

5.6 Blow Molding

Blow molding is used extensively to make bottles and other lightweight, hollow plastic parts. Two methods are used: injection blow molding and extrusion blow molding.

Injection blow molding is used primarily for small containers. The parison (molten-plastic pipe) or tube is formed by the injection of plasticized material around a hollow mandrel. While the material is still molten and still on the mandrel, it is transferred into the blowing mold where air is used to inflate it. Accurate threads may be formed at the neck.

In extrusion-type blow molding, parison is inflated under relatively low pressure inside a split-metal mold. The die closes, pinching the end and closing the top around the mandrel. Air enters through the mandrel and inflates the tube until the plastic contacts the cold wall, where it solidifies. The mold opens, the bottle is ejected, and the tailpiece falls off.

5.7 Thermoforming

Thermoforming refers to heating a sheet of plastic material until it becomes soft and pliable and then forming it under vacuum, by air pressure, or between matching mold halves.

5.8 Reinforced-Plastic Molding

Reinforced plastics generally refers to polymers that have been reinforced with glass fibers. Other materials used are asbestos, sisal, synthetic fibers such as nylon and polyvinyl chloride, and cotton fibers. High-strength composites using graphite fibers are now commercially available with moduli of 50,000,000 psi (344,700,000 MPa) and tensile strengths of about 300,000 psi (2,068,000 MPa). They are as strong as or stronger than the best alloy steels and are lighter than aluminum.

5.9 Forged-Plastic Parts

The forging of plastic materials is a relatively new process. It was developed to shape materials that are difficult or impossible to mold and is used as a low-cost solution for small production runs.

The forging operation starts with a blank or billet of the required shape and volume for the finished part. The blank is heated to a preselected temperature and transferred to the forging dies, which are closed to deform the work material and fill the die cavity. The dies are kept in the closed position for a definite period of time, usually 15–60 s. When the dies are opened, the finished forging is removed. Since forging involves deformation of the work material in a heated and softened condition, the process is applicable only to thermoplastics.

6 POWDER METALLURGY

In powder metallurgy (P/M), fine metal powders are pressed into a desired shape, usually in a metal die and under high pressure, and the compacted powder is then heated (sintered), with a protective atmosphere. The density of sintered compacts may be increased by repressing. Repressing is also performed to improve the dimensional accuracy, either concurrently or subsequently, for a period of time at a temperature below the melting point of the major constituent. P/M has a number of distinct advantages that account for its rapid growth in recent years, including (1) no material is wasted, (2) usually no machining is required, (3) only semiskilled labor is required, and (4) some unique properties can be obtained, such as controlled degrees of porosity and built-in lubrication.

A crude form of powder metallurgy appears to have existed in Egypt as early as 3000 BC, using particles of sponge iron. In the nineteenth century, P/M was used for producing platinum and tungsten wires. However, its first significant use related to general manufacturing was in Germany, following World War I, for making tungsten carbide cutting-tool tips. Since 1945 the process has been highly developed, and large quantities of a wide variety of P/M products are made annually, many of which could not be made by any other process. Most are under 2 in. (50.8 mm) in size, but many are larger, some weighing up to 50 lb (22.7 kg) and measuring up to 20 in. (508 mm).

Powder metallurgy normally consists of four basic steps:

1. Producing a fine metallic powder
2. Mixing and preparing the powder for use
3. Pressing the powder into the desired shape
4. Heating (sintering) the shape at an elevated temperature

Other operations can be added to obtain special results.

The pressing and sintering operations are of special importance. The pressing and repressing greatly affect the density of the product, which has a direct relationship to the strength properties. Sintering strips contaminants from the surface of the powder particles, permitting diffusion bonding to occur and resulting in a single piece of material. Sintering usually is done in a controlled, inert atmosphere, but sometimes it is done by the discharge of spark through the powder while it is under compaction in the mold.

6.1 Properties of P/M Products

Because the strength properties of powder metallurgy products depend on so many variables—type and size of powder, pressing pressure, sintering temperature, finishing treatments, and so on—it is difficult to give generalized information. In general, the strength properties of products that are made from pure metals (unalloyed) are about the same as those made from the same wrought metals. As alloying elements are added, the resulting strength properties of P/M products fall below those of wrought products by varying, but usually substantial, amounts. The ductility usually is markedly less, as might be expected because of the lower density. However, tensile strengths of 40,000–50,000 psi (275.8–344.8 MPa) are common, and strengths above 100,000 psi (689.5 MPa) can be obtained. As larger presses and forging combined with P/M preforms are used, to provide greater density, the strength properties of P/M materials will more nearly equal those of wrought materials. Coining can also be used to increase the strength properties of P/M products and to improve their dimensional accuracy.

7 SURFACE TREATMENT

Products that have been completed to their proper shape and size frequently require some type of surface finishing to enable them to satisfactorily fulfill their function. In some cases, it is necessary to improve the physical properties of the surface material for resistance to penetration or abrasion.

Surface finishing may sometimes become an intermediate step in processing. For instance, cleaning and polishing are usually essential before any kind of plating process. Another important need for surface finishing is for corrosion protection in a variety of environments. The type of protection provided will depend largely on the anticipated exposure, with due consideration to the material being protected and the economic factors involved.

Satisfying the above objectives necessitates the use of many surface-finishing methods that involve chemical change of the surface; mechanical work affecting surface properties, cleaning by a variety of methods, and the application of protective coatings organic and metallic.

7.1 Cleaning

Few, if any, shaping and sizing processes produce products that are usable without some type of cleaning unless special precautions are taken. Figure 22 indicates some of the cleaning methods available. Some cleaning methods provide multiple benefits. Cleaning and finish improvements are often combined. Probably of even greater importance is the combination of corrosion protection with finish improvement, although corrosion protection is more often a second step that involves coating an already cleaned surface with some other material or chemical conversion.

Liquid and Vapor Baths

Liquid and Vapor Solvents. The most widely used cleaning methods make use of a cleaning medium in liquid or vapor form. These methods depend on a solvent or chemical action between the surface contaminants and the cleaning material.

Petroleum Solvents. Among the more common cleaning jobs required is the removal of grease and oil deposited during manufacturing or intentionally coated on the work to provide protection. One of the most efficient ways to remove this material is by use of solvents that dissolve the grease and oil but have no effect on the base metal. Petroleum derivatives, such as Stoddard solvent and kerosene, are common for this purpose, but, since they introduce some danger of fire, chlorinated solvents, such as trichlorethylene, that are free of this fault are sometimes substituted.

Conditioned Water. One of the most economical cleaning materials is water. However, it is seldom used alone, even if the contaminant is fully water soluble, because the impurity of the water itself may contaminate the work surface.

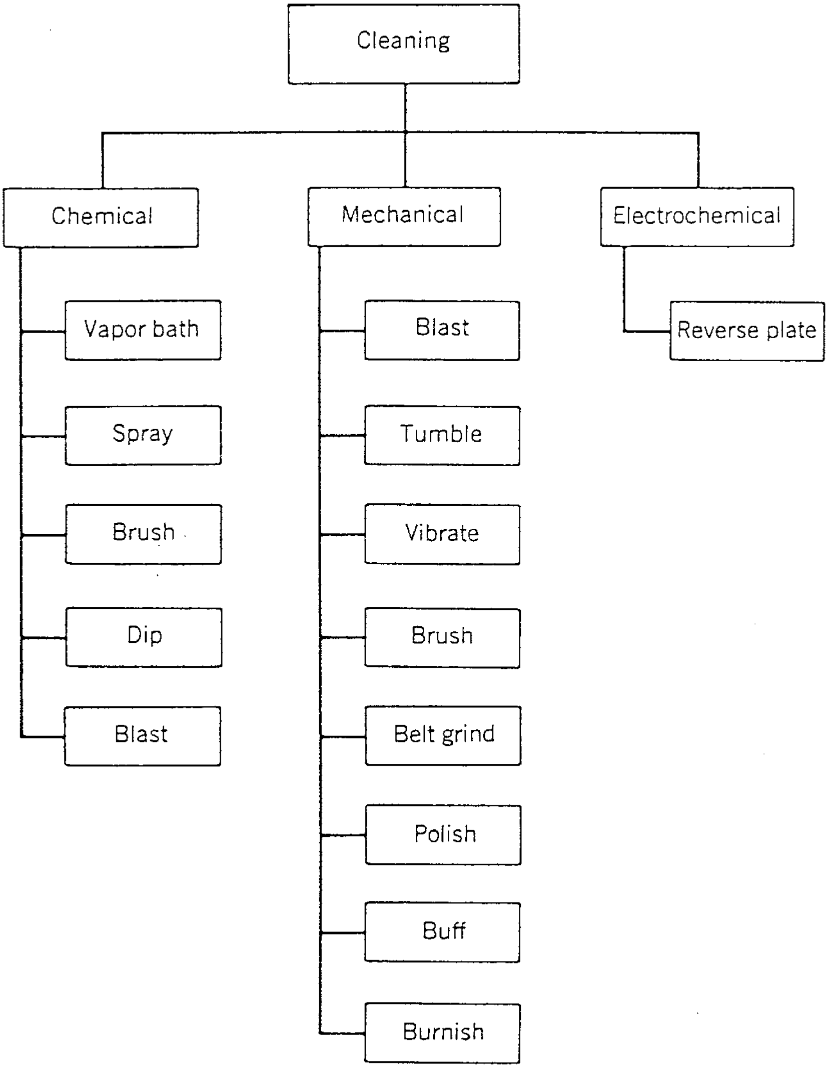

Fig. 22 Cleaning methods.

Depending on its use, water is treated with various acids and alkalies to suit the job being performed.

Pickling. Water containing sulfuric acid in a concentration from about 10–25% and at a temperature of approximately 149°F (65°C) is commonly used in a process called *pickling* for removal of surface oxides or scale or iron and steel.

Mechanical Work Frequently Combined with Chemical Action. Spraying, brushing, and dipping methods are also used with liquid cleaners. In nearly all cases, mechanical work to cause surface film breakdown and particle movement is combined with chemical and solvent action. The mechanical work may be agitation of the product, as in dipping, movement of the cleaning agent, as in spraying, or use of a third element, as in rubbing brushing. In some applications, sonic or ultrasonic vibrations are applied to either the solution or the workpieces to speed the cleaning action. Chemical activity is increased with higher temperatures and optimum concentration of the cleaning agent, both of which must in some cases be controlled closely for efficient action.

Blasting

The term *blasting* is used to refer to all those cleaning methods in which the cleaning medium is accelerated to high velocity and impinged against the surface to be cleaned. The high velocity may be provided by air or water directed through a nozzle or by mechanical means with a revolving slinger. The cleaning agent may be either dry or wet solid media, such as sand, abrasive, steel grit, or shot, or may be liquid or vapor solvents combined with abrasive material. In addition to cleaning, solid particles can improve finish and surface properties of the material on which they are used. Blasting tends to increase the surface area and thus set up compressive stresses that may cause a warping of thin sections, but in other cases, it may be very beneficial in reducing the likelihood of fatigue failure. When used for the latter purpose, the process is more commonly known as *shot peening.*

Water Slurries. Liquid or vaporized solvents may, by themselves, be blasted against a surface for high-speed cleaning of oil and grease films with both chemical and mechanical action. Water containing rust-inhibiting chemicals may carry, in suspension, fine abrasive particles that provide a grinding cutting-type action for finish improvement along with cleaning. The blasting method using this medium is commonly known as *liquid honing.*

Abrasive Barrel Finishing

Barrel finishing, rolling, tumbling, and *rattling* are terms used to describe similar operations that consist of packing parts together with some cleaning media in a cylinder or drum, which can be rotated to cause movement among them. The media may be abrasive (either fine or coarse); metal stars, slugs, or balls; stones; wood chips; sawdust; or cereals. The work may be done wet or dry, depending on the materials being worked with, the kind of surface finish desired, and the kind of equipment available.

Wire Brushing

A number of cleaning operations can be quickly and easily performed by use of a high-speed rotating wire brush. In addition to cleaning, the contact rubbing of the wire ends across the work surface produce surface improvement by a burnishing-type action. Sharp edges and burrs can be removed.

Abrasive Belt Finishing

Continuous fabric belts coated with abrasive can be driven in several kinds of machines to provide a straight-line cutting motion for grinding, smoothing, and polishing work surfaces. Plane surfaces are the most common surfaces worked on with fabric belts.

Polishing

The term *polishing* may be interpreted to mean any nonprecision procedure providing a glossy surface but is most commonly used to refer to a surface-finishing process using a flexible abrasive wheel. The wheels may be constructed of felt or rubber with an abrasive band, of multiple coated abrasive disks, of leaves of coated abrasive, of felt or fabric to which loose abrasive is added as needed, or of abrasives in a rubber matrix.

Buffing

About the only difference between buffing and polishing is that, for buffing, a fine abrasive carried in wax or a similar substance is charged on the surface of a flexible level.

Electropolishing

If a workpiece is suspended in an electrolyte and connected to the anode in an electrical circuit, it will supply metal to the electrolyte in a reverse plating process. Material will be removed faster from the high spots of the surface than from the depressions and will thereby increase the average smoothness. The cost of the process is prohibitive for very rough surfaces because larger amounts of metal must be removed to improve surface finish than would be necessary for the same degree of improvement by mechanical polishing. Electropolishing is economical only for improving a surface that is already good or for polishing complex and irregular shapes, the surfaces of which are not accessible to mechanical polishing and buffing equipment.

7.2 Coatings

Many products, particularly those exposed to view and those subject to change by the environment with which they are in contact, need some type of coating for improved appearance or for protection from chemical attack. The need for corrosion protection for maintenance and appearance is important. In addition to change of appearance, loss of actual material, change of dimensions, and decrease of strength, corrosion may be the cause of eventual loss of service or failure of a product. Material that must carry loads in structural applications, especially when the loads are cyclic in nature, may fail with fatigue if corrosion is allowed to take place. Corrosion occurs more readily in highly stressed material, where it attacks grain boundaries in such a way as to form points of stress concentration that may be nuclei for fatigue failure.

Harness and wear resistance, however, can be provided on a surface by plating with hard metals. Chromium plating of gauges and other parts subject to abrasion is frequently used to increase their wear life. Coatings of plastic material and asphaltic mixtures are sometimes placed on surfaces to provide sound deadening. The additional benefit of protection from corrosion is usually acquired at the same time.

Plastics of many kinds, mostly of the thermoplastic type because they are easier to apply and also easier to remove later if necessary, are used for mechanical protection. Highly polished material may be coated with plastic, which may be stripped off later, to prevent abrasion and scratches during processing.

It is common practice to coat newly sharpened cutting edges of tools by dipping them in thermoplastic material to provide mechanical protection during handling and storage.

Organic Coatings

Organic coatings are used to provide pleasing colors, to smooth surfaces, to provide uniformity in both color and texture, and to act as a protective film for control of corrosion. Organic resin coatings do not ordinarily supply any chemical-inhibiting qualities. Instead, they merely provide a separating film between the surface to be protected and the corrosive environment. The important properties, therefore, are continuity, permeability, and adhesion characteristics.

Paints, Varnishes, and Enamels

Paints. Painting is a generic term that has come to mean the application of almost any kind of organic coating by any method. Because of this interpretation, it is also used generally to describe a broad class of products. As originally defined and as used most at present, paint is a mixture of pigment in a drying oil. The oil serves as a carrier for the pigment and in addition creates a tough continuous film as it dries. Drying oils, one of the common ones of which is linseed oil, become solid when large surface areas are exposed to air. Drying starts with a chemical reaction of oxidation. Nonreversible polymerization accompanies oxidation to complete the change from liquid to solid.

Varnish. Varnish is a combination of natural or synthetic resins and drying oil, sometimes containing volatile solvents as well. The material dries by a chemical reaction in the drying oil to a clear or slightly amber-colored film.

Enamel. Enamel is a mixture of pigment in varnish. The resins in the varnish cause the material to dry to a smoother, harder, and glossier surface than is produced by ordinary paints. Some enamels are made with thermosetting resins that must be baked for complete dryness. These baking enamels provide a toughness and durability not usually available with ordinary paints and enamels.

Lacquers

The term *lacquer* is used to refer to finishes consisting of thermoplastic materials dissolved in fast-drying solvents. One common combination is cellulose nitrate dissolved in butyl acetate. Present-day lacquers are strictly air-dried and form films very quickly after being applied, usually by spraying. No chemical change occurs during the hardening of lacquers; consequently, the dry film can be redissolved in the thinner. Cellulose acetate is used in place of cellulose nitrate in some lacquers because it is nonflammable. Vinyls, chlorinated hydrocarbons, acrylics, and other synthetic thermoplastic resins are also used in the manufacture of lacquers.

Vitreous Enamels

Vitreous, or porcelain, enamel is actually a thin layer of glass fused onto the surface of a metal, usually steel or iron. Shattered glass, ball milled in a fine particle size, is called *frit.* Frit is mixed with clay, water, and metal oxides, which produce the desired color, to form a thin slurry called *slip*. This is applied to the prepared metal surface by dipping or spraying and, after drying, is fired at approximately 1470°F (800°C) to fuse the material to the metal surface.

Metallizing

Metal spraying, or metallizing, is a process in which metal wire or powder is fed into an oxyacetylene heating flame and then, after melting, is carried by high-velocity air to be impinged against the work surface. The small droplets adhere to the surface and bond together to build up a coating.

Vacuum Metallizing

Some metals can be deposited in very thin films, usually for reflective or decorative purposes, as a vapor deposit. The metal is vaporized in a high-vacuum chamber containing the parts to be coated. The metal vapor condenses on the exposed surfaces in a thin film that follows the surface pattern. The process is cheap for coating small parts, considering the time element only, but the cost of special equipment needed is relatively high.

Aluminum is the most used metal for deposit by this method and is used frequently for decorating or producing a mirror surface on plastics. The thin films usually require mechanical protection by covering with lacquer or some other coating material.

Hot-Dip Plating

Several metals, mainly zinc, tin, and lead, are applied to steel for corrosion protection by a hot-dip process. Steel in sheet, rod, pipe, or fabricated form, properly cleansed and fluxed, is immersed in molten plating metal. As the work is withdrawn, the molten metal that adheres solidifies to form a protective coat. In some of the large mills, the application is made continuously to coil stock that is fed through the necessary baths and even finally inspected before being recoiled or cut into sheets.

Electroplating

Coatings of many metals can be deposited on other metals, and on nonmetals when suitably prepared, by electroplating. The objectives of plating are to provide protection against corrosion, to improve appearance, to establish wear- and abrasion-resistant surfaces, to add material for dimensional increase, and to serve as an intermediate step of multiple coating. Some of the most common metals deposited in this way are copper, nickel, cadmium, zinc, tin, silver, and gold. The majority are used to provide some kind of corrosion protection but appearance also plays a strong part in their use.

Temporary Corrosion Protection

It is not uncommon in industry for periods of time, sometimes quite long periods, to elapse between manufacture, assembly, shipment, and use of parts. Unless a new processing schedule can be worked out, about the only cure for the problem is corrosion protection suitable for the storage time and exposure. The coatings used are usually nondrying organic materials, called *shushing compounds,* that can be removed easily. The two principal types of compounds used for this purpose are petroleum-based materials, varying from extremely light oils to semisolids, and thermoplastics. The most common method of application of shushing compounds for small parts is by dipping. Larger parts that cannot be handled easily may be sprayed, brushed, or flow coated with the compound.

7.3 Chemical Conversions

A relatively simple and often fully satisfactory method for protection from corrosion is by conversion of some of the surface material to a chemical composition that resists from the environment. These converted metal surfaces consist of relatively thin [seldom more than 0.001 in. (0.025 mm) thick] inorganic films that are formed by chemical reaction with the base material. One important feature of the conversion process is that the coatings have little effect on the product dimensions.

Anodizing

Aluminum, magnesium, and zinc can be treated electrically in a suitable electrolyte to produce a corrosion-resistant oxide coating. The metal being treated is connected to the anode in the circuit, which provides the name *anodizing* for the process. Aluminum is commonly treated by anodizing that produces an oxide film thicker than, but similar to, that formed naturally with exposure to air. Anodizing of zinc has very limited use. The coating produced on magnesium is not as protective as that formed on aluminum, but does provide some protective value and substantially increases protection when used in combination with paint coatings.

Chromate Coatings

Zinc is usually considered to have relatively good corrosion resistance. This is true when the exposure is to normal outdoor atmosphere where a relatively thin corrosion film forms. Contact with either highly aerated water films or immersion in stagnant water containing little oxygen causes uneven corrosion and pitting. The corrosion products of zinc are less dense than the base material, so that heavy corrosion not only destroys the product appearance, but also may cause malfunction by binding moving parts. Corrosion of zinc can be substantially slowed by the production of chromium salts on its surface. The corrosion resistance of magnesium alloys can be increased by immersion of anodic treatment in acid baths containing dichromates. Chromate treatment of both zinc and magnesium improves corrosion resistance but is used also to improve adhesion of paint.

Phosphate Coatings

Phosphate coatings, used mostly on steel, result from a chemical reaction of phosphoric acid with the metal to form a nonmetallic coating that is essentially phosphoric salts. The coating is produced by immersing small items or spraying large items with the phosphating solution. Phosphate surfaces may be used alone for corrosion resistance, but their most common application is as a base for paint coatings. Two of the most common application methods are called *parkerizing* and *bonderizing*.

Chemical Oxide Coatings

A number of proprietary blacking processes, used mainly on steel, produce attractive black oxide coatings. Most of the processes involve the immersing of steel in a caustic soda solution, heated to about 300°F (150°C) and made strongly oxidizing by the addition of nitrites or nitrates. Corrosion resistance is rather poor unless improved by application of oil, lacquer, or wax. As in the case of most of the other chemical conversion procedures, this procedure also finds use as a base for paint finishes.

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CHAPTER 32

PLASTIC PARTS PROCESSING I

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1 INTRODUCTION

The processing of plastics materials is characterized by a wide variety of techniques, including (1) techniques involving the continuous manufacture of a product with a uniform cross section, such as extrusion, extrusion covering, film blowing, and calendering; (2) techniques involving the shaping of a deformable polymer preform against a mold surface, including sheet thermoforming and blow molding; (3) techniques involving the gradual buildup of a polymer layer against a mold surface, such as coating and rotational molding; and (4) techniques involving the filling of a mold cavity, including casting, compression molding, transfer molding, injection molding, and reaction injection molding. This chapter discusses each of these techniques, including the basic principles, equipment, tooling, and auxiliary apparatus involved in the application of each technique.

2 EXTRUSION

Principle

The extrusion process consists of continuously shaping a fluid polymer through the orifice of a die and subsequently solidifying it into a product. The feed

Handbook of Materials Selection. Edited by Myer Kutz

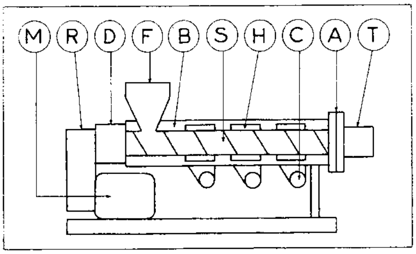

Fig. 1 Single-screw extruder: M, electric motor; R, speed reduction device; D, thrust bearing; F, feed hopper; B, barrel; S, screw; H, heater; C, cooler; A, adapter; T, tool (die). (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

material, usually a thermoplastic in powder or pellet form, is heated to a fluid state and forced through the die using a screw extruder. The extruded product is solidified by cooling after it exits the die.

Equipment

A conventional single-screw extruder consists of several important elements, as illustrated in Fig. 1. The screw rotates within a cylindrical barrel. The material enters the extruder through a feed hopper and exits through the die, which is fitted to the extruder through an adapter. The mechanical drive system consists of a variable-speed electric motor and a speed reduction device. The barrel of a conventional extruder has a circular cylindrical bore. Extruders are generally rated by the barrel bore diameter. This diameter can be as low as about $\frac{3}{4}$ in. (20 mm) for laboratory extruders and from 2 to 6 in. (65 to 150 mm) for commercial extruders.

The simple screw shown in Fig. 2 consists of three zones corresponding to the three stages in the process. Zone $\boxed{S}$ is primarily involved with the conveying of the solid material and features a relatively deep channel (depth H_s). Zone $\boxed{M}$ is associated primarily with the metering and pressurizing (1000–3000 psi or 70–200 atm) of the molten material and features a shallower channel (depth H_m). Most melting takes place over the transition zone $\boxed{T}$. The ratio of the total screw length to the barrel diameter (L/D) is usually referred to as the "screw aspect ratio" and is usually in the range 20–30. The ratio of channel depths H_s/H_m is called the "compression ratio" and is typically in the range from 2 to 6.

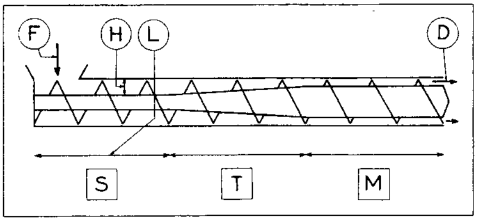

Fig. 2 Simple extruder screw: $\boxed{S}$, solid-conveying zone; $\boxed{T}$, transition zone; $\boxed{M}$, melt-conveying zone; H, channel depth; L, zone length. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

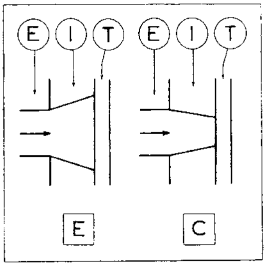

Fig. 3 Plate die configurations: $\boxed{\text{E}}$, expanding transition; $\boxed{\text{C}}$, contracting transition; E, extruder exit; I, transition piece (preland); T, die plate (land). (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

Tooling

The shaping tool associated with extrusion is usually called the *die*. Widely different die designs are associated with filaments, profile products, tubes or pipes, and films or sheets. *Filaments* are often made from plate dies featuring multiple orifices with streamlined (tapered) entrances. Profiles without hollow sections are also made from plate dies, as illustrated in Fig. 3. The formation of a hollow extrudate, such as a tube or a pipe, requires the use of a mandrel to form the inner surface, while a bushing forms the outer surface, as shown in Fig. 4.

Auxiliaries

Although the die is the major element in the formation of the extruded product, downstream auxiliaries are often essential to meeting product specifications while maximizing output. Auxiliaries include cooling, sizing, post forming, and haul-off, as well as cutting and/or winding devices. Cooling is usually achieved through air convection in the case of thin extrudates or through water convection (immersion) in a tank trough. Evaporation spray, a more efficient method, is often used for pipes and profiles. Sizing depends on the shape and dimensional specifications of the products, with the thickness of extruded sheets, films, pipes, and other configurations typically monitored with a variety of gauges.

Materials

Practically all thermoplastics can be processed by extrusion, but in order for the extrudates to maintain their shape until they solidify, extrusion grades tend to

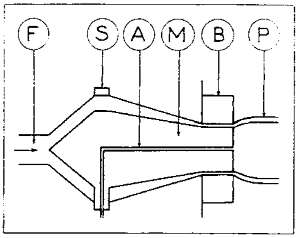

Fig. 4 Tubing die: F, feed; S, spider; A, air channel; M, mandrel; B, bushing; P, product. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

have relatively high molecular weights associated with high viscosity and melt strength. Conventional rubbers and, to a much smaller extent, thermosets, are extruded to form profiles, for example, but they must be subsequently cured.

Products

- Fibers and monofilaments are extruded from a number of thermoplastics.
- Profiles are extruded in a wide variety of shapes, from flat strips or simple channels to very complex shapes, including hollow sections.
- Flat products are usually subdivided into films, when thinner than 0.010 in., and sheets above that.

3 EXTRUSION COVERING

Principle

The expression "extrusion covering" is used here to describe a process that completely surrounds a continuous substrate with a cover of polymeric material. Its most common use is for the formation of an insulating layer or a protective jacket on power or communication electrical wires and cables.

Equipment

An extrusion covering line, shown in Fig. 5, comprises a substrate pay-off (input) system, a substrate preheater [typically to around 100°C (≈200°F)], an extruder fitted with an appropriate extrusion covering tool, a cooling device, and a take-up (output) system for the product.

Tooling

In its simplest form for the case of wire coating, an extrusion covering die (Fig. 6), mounted with its extrusion axis perpendicular to the extruder axis (crosshead die), comprises a tapered guider or core tube for the substrate, and a suitable channel to direct the covering fluid polymer, from the extruder exit to an annular gap between a bushing and the continuously moving substrate.

Materials

Plasticized polyvinyl chloride (PPVC) and polyethylene (PE) are the most widely used thermoplastic covering materials, but other plastics such as polyamides, fluoropolymers, and cellulosics are used for wire insulation.

Products

Wire and cable coating, sheathing, and jacketing is the most common application for extrusion covering.

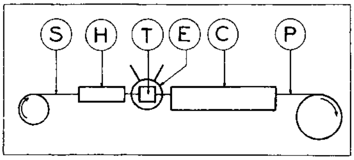

Fig. 5 Extrusion covering line: S, substrate; H, preheater; T, tool (die); E, extruder; C, cooler; P, product. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

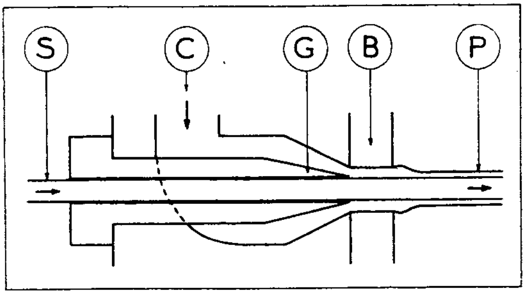

Fig. 6 Extrusion covering die: S, substrate; C, covering material; G, substrate guider; B, bushing; P, product. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

4 FILM BLOWING

Principle

The film-blowing process basically consists of extruding a tube of molten thermoplastic and continuously inflating it to several times its initial diameter, to form a thin tubular product that can be used directly or slit to form a flat film.

Equipment

As illustrated in Fig. 7, the first element of a conventional film-blowing line is the extruder, which supplies one, and sometimes two or more, tubular dies, described subsequently in the section on tooling. The molten tube, as it emerges from the die, is subjected to both a moderate internal air pressure by way of an air inlet running through the die mandrel, and a longitudinal force exerted by take-off rolls. The tube is simultaneously cooled by air from an external air ring. The molten tube gradually deforms into a stable solid cylindrical bubble beyond the frost (freeze) line. The bubble is gradually flattened in the collapsing device (tent frame), which consists of a pair of converging, ladderlike sets of idler rolls or wood slats, and beyond the pinch (nip) rolls it is handled as a thin flat product.

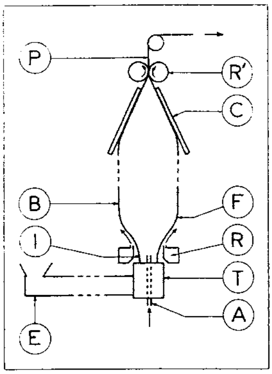

Fig. 7 Film-blowing line: E, extruder; I, molten tube; B, bubble; P, product; R′, pinch/take-off rolls; C, collapsing device; F, freeze line; R, cooling air ring; T, tool (die); A, air inlet. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

Startup of the film-blowing operation normally involves the pulling of the uninflated extruded tube, with the help of a cable, until it becomes pinched between the nip rolls. Internal air pressure is subsequently applied to form the bubble. The blow-up ratio (BUR), defined as the ratio of bubble to die diameters (or circumferences), is normally in the range 1.5–4, with larger values requiring high melt strengths. The air pressure in the bubble, which is responsible for the blowing, is normally in the range 0.05–0.2 atm.

Tooling

The extrusion die that produces the tube of molten polymer is the key element of the process. Crosshead dies of the bottom-fed/spider type tend to cause problems with weld lines, while it is difficult to obtain flowrate uniformity with side-fed/spiderless types. Modern film-blowing dies are increasingly of the bottom-fed/spiral mandrel type. Dies for coextruded, multilayer blown films feature several concentric spiral groove systems with associated clearances, leading to individual prelands and the common die land.

Auxiliaries

While the normal output of a film-blowing line is a tubular, lay-flat product, the introduction of slitters, cutters, punches, gusseting and sealing devices, and other instruments can allow in-line production of slit-open or lay-flat products, and gusseted products, as well as a variety of bags.

Materials

Polyethylenes of various types [low-density polyethylene (LDPE), linear low-density polyethylene (LLDPE), high molecular-weight high-density polyethylene (HMWHDPE), etc.] are by far the most common film-blowing resins.

Products

Films produced by the film-blowing process are widely used in many areas. Thick-gauge, heavy-duty films are often in the range 0.004–0.008 in. Films are also widely used for a variety of packaging applications. Blown films often find applications as shrink-wrap films. Multilayer, coextruded blown films are increasingly used for barrier packaging.

5 CALENDERING

Principle

The calendering process, depicted in Fig. 8, is used for the fast production of flat films or sheets of plastics or rubbers. A suitable amount of plasticated material is regularly fed between two counterrotating heated cylinders (rolls) (R and R′). The material is entrained at the surface of the rolls and squeezed in the wedge, and a layer of a thickness corresponding approximately to the shortest distance between roll surfaces (gap) is formed through the nip and entrained on the surface of one of the rolls (R′).

Equipment, Tooling, and Auxiliaries

The diameter of calendaring rolls is generally in the range 40–90 cm and their length is generally in the range 1–2.5 m; this corresponds to length: diameter ratios between 2 and 3. Calendering units generally feature 3–5 rolls (2–4 nips),

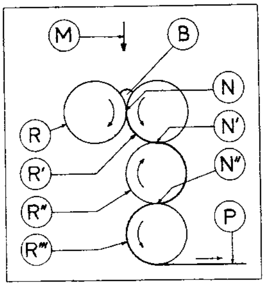

Fig. 8 Calendering process: R, R′, feed rolls; R″, R‴, gauge/finishing rolls; M, feed material; B, material bank; N, feed nip; N′, intermediate nip; N″, gauge nip; P, product. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

arranged in a variety of configurations (roll axis pattern or sheet path) as shown in Fig. 9 The last roll, or pair of rolls, in the calendering stack determines the surface finish of the product.

Materials and Products

Vinyl thermoplastics (PVC and copolymers) and vulcanizable rubbers are the most commonly calendered materials. Film thickness is not normally below about 0.05 mm. Sheets can be as thick as about 6 mm. Examples of applications include vinyl films made into products such as baby pants, inflatable toys, shower curtains, tablecloths, pool liners, wall covering, and veneered panels as well as vinyl sheets for credit cards, blister packaging, floor tiles, and floor covering (linoleum).

6 SHEET THERMOFORMING

Principle

Sheet thermoforming, or simply thermoforming, involves the heating of a flat thermoplastic sheet to a softened state (above the glass transition temperature T_g for noncrystallizing thermoplastics or near the melting temperature T_m for crystallizing ones), followed by the deformation (forming) of the softened sheet into a desired shape by pneumatic or mechanical means, and finally its solidification into this shape by cooling. Thermoforming transforms a flat sheet of uniform thickness into a contoured product of variable thickness resulting from the nonuniform biaxial stretching. Figure 10 illustrates this concept.

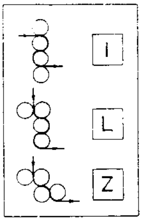

Fig. 9 Calendering rolls configurations: $\boxed{\text{I}}$, vertically aligned; $\boxed{\text{L}}$, inverted L; $\boxed{\text{Z}}$, Z-like. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

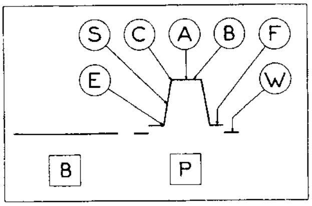

Fig. 10 Typical cuplike product: $\boxed{\text{B}}$, blank; $\boxed{\text{P}}$, product; E, edge; S, side; C, corner; A, apex; B, bottom; F, flange; W, web. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

Thermoforming normally involves a one-surface (single-surface) tool, and it is convenient to distinguish between negative (female, cavity) tools, which are basically concave and into which the sheet is formed (drawn), and positive (male, plunger) tools, which are basically convex and over which the sheet is formed (draped). Pneumatic deformation is most commonly done by vacuum (suction). Mechanical deformation tends to be used primarily for preforming or prestretching, before final vacuum or air pressure forming. The simplest thermoforming technique is perhaps that referred to as *straight vacuum thermoforming*, which is illustrated in Fig. 11.

Equipment

The simplest commercial type is probably the single-station shuttle thermoformer, shown in Fig. 12. The two-station (double-end) shuttle thermoformer is a variation that uses a single oven with two workstations.

Tooling

Tools for thermoforming are not normally subjected to high stresses since the pressure differential is often limited to 1 atm in vacuum forming or 3–4 atm in pressure forming. They are often made from cast or machined aluminum for small and medium sizes. Epoxy and polyester resins, with fiber reinforcement and a heat-conductive filler, are suitable for the relatively slow production of large parts. Tools are generally water-cooled to temperature in the range 40–95°C, while mechanical assist devices (plugs) are usually maintained at a some-

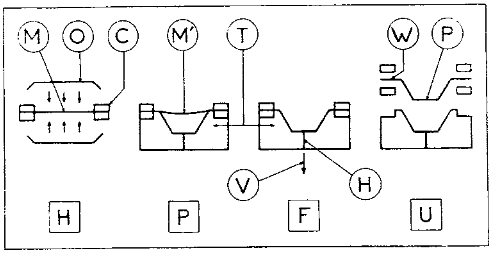

Fig. 11 Straight vacuum thermoforming: $\boxed{\text{H}}$, heating; $\boxed{\text{P}}$, positioning; $\boxed{\text{F}}$, forming; $\boxed{\text{U}}$, unloading; M, blank; O, oven; C, clamp; M′, softened blank; T, tool (mold); V, vacuum; H, vacuum hole(s); W, web; P, product. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

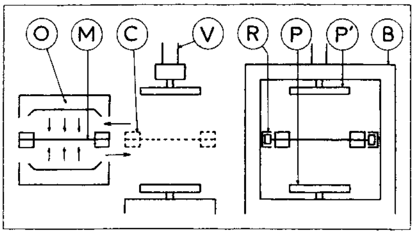

Fig. 12 Single-station shuttle thermoformer: O, oven; M, blank; C, clamp; V, air cylinder; R, rack; P, P′, platens; B, frame. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

what higher temperature (60–150°C). Vacuum is applied through fine holes, slots, or ports that do not leave significant marks on products.

Auxiliaries

Heating of the sheet stock (blank) is most commonly carried out with radiant electric heaters (nichrome wire, calrod, ceramic, quartz, etc.), which correspond to electric power densities around 15–25 kW/m^2. After forming, products must be separated from the surrounding web by a trimming operation.

Materials

Noncrystallizing thermoplastics are normally easier to thermoform then crystallizing ones because of their highly elastic behavior over a fairly wide range of temperature above T_g. They include styrenics such as high-impact (HI) or crystalline polystyrene (PS), and copolymers such as acrylonitrile-butadiene-styrene (ABS).

Products

Products made by sheet thermoforming fall into two distinct categories. Small products made in high-output machines, often of the roll-fed, in-line type, using multicavity tools, are associated primarily with packaging and disposable items such as those used in the food industry and for medical applications. Larger products are generally made from cut sheets at much slower rates.

7 BLOW MOLDING

Principle

The basic principle of the blow-molding process is to inflate a softened thermoplastic hollow preform against the cooled surface of a closed mold, where the material solidifies into a hollow product. The extrusion blow-molding (EBM) process, depicted in Fig. 13, involves an extruder fitted with a die or die head that produces the preform by extruding downward a tube of thermoplastic generally called preform or parison at stage $\boxed{E}$. While the preform is still soft, it is then pinched (stage $\boxed{P}$) between the two halves of a mold; the bottom is pinched shut and sealed, while the top is pinched around a metal tube or blow pin protruding from the die core. At the following stage $\boxed{B}$, the soft preform is blown against the cooled mold surfaces by air injected through the blow pin.

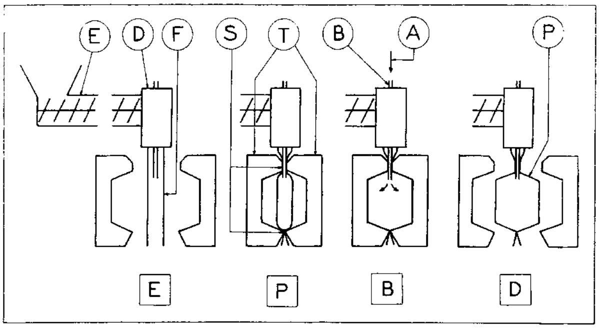

Fig. 13 Extrusion blow molding: $\boxed{\text{E}}$, extrusion; $\boxed{\text{P}}$, pinching; $\boxed{\text{B}}$, blowing; $\boxed{\text{D}}$, demolding; E, extruder; D, die; F, preform; S, squeezed area; T, tool; B, blow pin; A, air pressure; P, product. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

After the thermoplastic material has solidified, the mold is opened and the product is pulled out (demolded) at stage $\boxed{\text{D}}$.

The injection blow-molding (IBM) process, illustrated in Fig. 14, first involves an injection stage $\boxed{\text{I}}$, during which the thermoplastic material is injected into an injection mold featuring two important elements, a split neck ring and a core pin. Upon demolding $\boxed{\text{D}}$, the preform remains attached to the neck ring and core pin, but the injection mold is replaced by a blow mold (stage $\boxed{\text{P}}$). Air is subsequently injected through a valve in the core pin, to blow the soft preform against the cooled blow mold surface, to form the product. The core is retracted and the neck ring and the mold simultaneously split open to demold the product.

Equipment

Extrusion blow-molding (EBM) machines basically consist of two major elements: (1) the parison former, which comprises an extruder, a die, and sometimes a special accumulating device; and (2) the mold-handling device (mold carrier). Injection blow-molding (IBM) machines often feature the rotary system with stationary injection mold as well as blow mold and revolving hub, which transfers the preform from the injection station to the blowing station, and the product

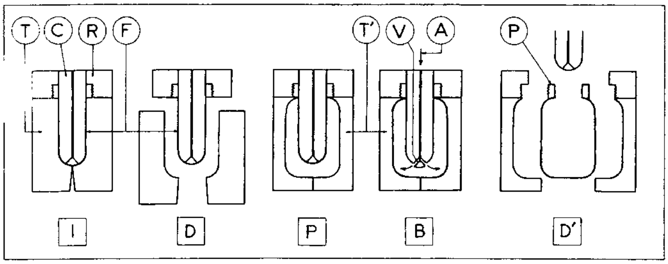

Fig. 14 Injection blow molding: $\boxed{\text{I}}$, preform injection; $\boxed{\text{D}}$, preform demolding; $\boxed{\text{P}}$, tool replacement; $\boxed{\text{B}}$, product blowing; $\boxed{\text{D}}'$, product demolding; T, injection tool, C, core pin; R, neck ring; F, preform; T′, blowing tool; V, air valve; A, air pressure; P, product. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

to the demolding station. Blowing pressures are often limited to normal plant air pressure (about 120 psi) and clamping forces are thus relatively small (typically about 1 ton for a bottle and about 50 tons for a shipping drum).

Tooling

Tooling for a specific extrusion blow-molded product involves the mold as well as the die to produce the parison. In the case of injection blow molding, an injection mold is needed for the preform, in addition to the blow mold. Blow molds are subjected to moderate pressures and clamping forces, compared to injection molds; they can thus be constructed more lightly, and aluminum is often used, which has advantages in terms of weight and thermal conductivity.

Auxiliaries

Extrusion blow molding produces a significant amount of process scrap, which is usually recycled after regrinding. Process scrap is most often associated with the flash resulting from the pinching operation. Such scrap can amount to as much as 25% for bottles and 50% for toys.

Materials

The process of blow molding requires a good stretchability of the softened thermoplastic (melt strength) over a reasonable temperature range. A relatively high molecular weight material is normally required, particularly for crystallizing resins. Polyethylene (PE) is, by far, the most widely used resin for large products. Polyvinyl chloride (PVC) is widely used for bottles. Thermoplastic polyester (PET) is primarily used in injection blow molding.

Products

Blow-molded products have certain inherent design limitations, concerning shapes and wall thickness in particular. While the blow-molding process normally yields one-piece, closed, hollow products, pairs of open, hollow products have been made by splitting closed moldings. Packaging is the major area of application of small to medium-size disposable blow-molded products. Figure 15 shows several typical blow-molded products.

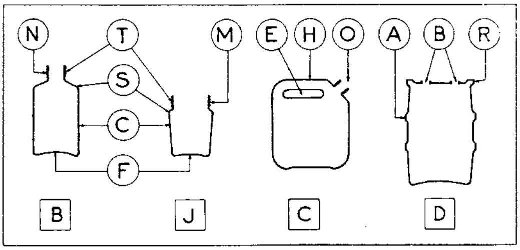

Fig. 15 Blow-molded products: B, bottle; J, jar; C, canister; D, drum; N, narrow neck; T, thread; S, shoulder; C, body; F, base; M, wide mouth; E, eye; H, handle; O, off-center neck; A, annular rib; B, bungs; R, ring. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

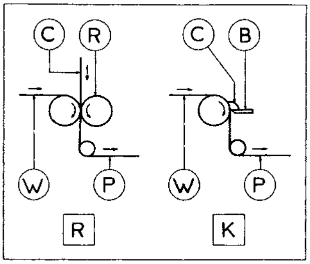

Fig. 16 Planar coating: $\boxed{R}$, roller technique; $\boxed{K}$, knife technique; W, web; C, coating material; R, pressure rolls; B, doctor blade; P, product. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

8 COATING

Coating may be defined as the formation (application) of a layer (coat) of polymer on a substrate; "planar coating," is used for flat film and sheet substrates and "contour coating," for general three-dimensional objects.

Planar Coating

A variety of flexible flat substrates (webs) are coated with polymers for a variety of reasons, including appearance, waterproofness, electrical insulation, and sealability. The adhesion between substrate and coat is always important, and various methods are used to favor a good bond.

Two basic techniques, as illustrated in Fig. 16, are used to form the coating and control its thickness. The roller (roll) technique $\boxed{R}$ squeezes the fluid coating material against the substrate web in the wedge (nip), between counterrotating pressure (nip) rolls. In the knife technique $\boxed{K}$, a blade (doctor blade) holds an excess of coating material, allowing the entrainment of a suitable amount through a small gap.

Contour Coating

Objects of complex shapes can be coated by dipping them in suitable fluids as shown in Fig. 17 or by fluidized-bed dip coating as illustrated in Fig. 18.

9 ROTATIONAL MOLDING

Principle

The process of rotational molding is depicted in Fig. 19. At the loading stage $\boxed{L}$, a suitable powdery thermoplastic material charge is introduced in the open

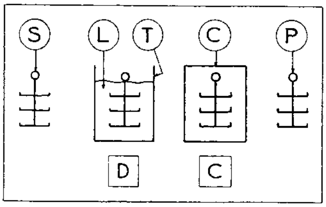

Fig. 17 Liquid dip coating: $\boxed{D}$, dipping; $\boxed{C}$, conditioning; S, substrate; L, coating liquid; T, tank; C, conditioning chamber; P, product. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

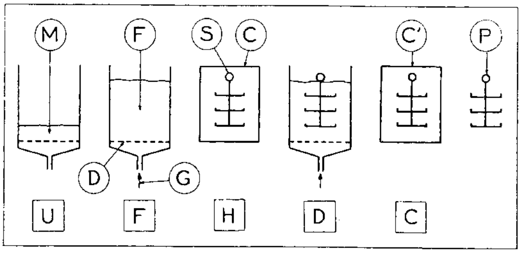

Fig. 18 Fluidized-bed dip coating: $\boxed{U}$, unfluidized bed; $\boxed{F}$, fluidized bed; $\boxed{H}$, substrate heating; $\boxed{D}$, dipping; $\boxed{C}$, conditioning; M, powdered coating material; F, fluidized particles; D, porous diaphragm; G, gas inlet; S, substrate; C, heating chamber; C′, conditioning chamber; P, product. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

mold (mold halves T and T′). The mold is then closed and mounted on a holding device, which permits its double, biaxial, rotation around two perpendicular axes (R and R′) to produce a tumbling action. A heating stage $\boxed{H}$ follows in which the exterior of the mold is subjected to a high temperature in a suitable chamber, while being roasted. Heat transfer through the mold wall eventually raises the temperature of the inner mold surface to a level sufficient to cause superficial melting of the thermoplastic particles and their sticking to the mold surface.

Equipment

The mold-handling device, which is capable of imparting double rotation, is the central element of rotational molding equipment. There are two major types of equipment: (1) the "shuttle cart system" and (2) the "swing/rotary arm system." The shuttle cart system, shown in Fig. 20, features a cart (wagon), incorporating the mold rotation device, which can be rolled in and out of the heating or cooling chamber (bay). The swing/rotary arm system, illustrated in Fig. 21, features arms or spindles that rotate in a horizontal plane around a hub.

Tooling

Molds for rotational molding are normally split (two-half, two-piece) negative molds, with built-in mechanical locking. The need for rapid heat transfer, and minimum weight to facilitate rotation, calls for relatively thin walls, which are acceptable in view of the low pressures involved. Large molds are generally

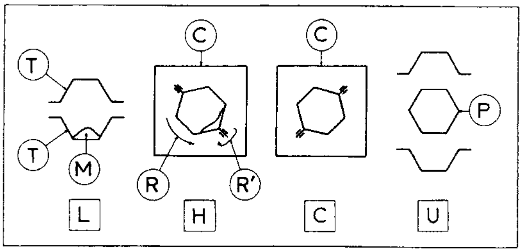

Fig. 19 Rotational molding process: $\boxed{L}$, loading; $\boxed{H}$, heating; $\boxed{C}$, cooling; $\boxed{U}$, unloading; T, T′, mold halves; M, material charge; R, R′, axes of rotation; C, conditioning chamber; P, product. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

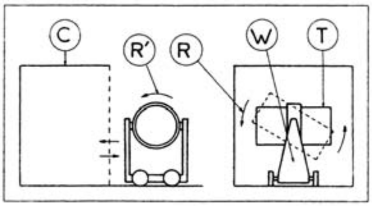

Fig. 20 Shuttle cart rotational molding: C, conditioning chamber; R′, rotation axis; R, tilting axis; W, cart; T, mold. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

fabricated from steel or sometimes aluminum in sheet form. Smaller molds are generally made from aluminum, which is light and heat conductive.

Materials

The nature of the process normally calls for the use of a relatively thin powder of a thermoplastic polymer. Particles featuring sharp edges and corners process better than purely spherical ones, probably because of the rapid softening of those areas. Polyethylene is widely used, particularly in its high-density form high-density polyethylene (HDPE). Polypropylene (PP) and polymethyleneoxide (PMO) are among other crystallizing thermoplastics used in this process.

Products

The part thickness, which is heat-transfer-controlled, can be quite uniform and is usually in the range of 3–6 mm. Corners and edges are often thicker as a result of concentrated heat transfer, and, conversely, insulated areas do not become covered with polymer, thus forming openings.

10 CASTING

Principle, Materials, and Products

Casting corresponds to the pouring or casting of the liquid resin system into a mold (gravity or atmospheric pressure casting), as illustrated in Fig. 22. In some cases, the chemical reaction that is taking place during a casting process converts a low-molecular-weight monomer into a high-molecular-weight thermoplastic; the most common examples are acrylics and polyamides. Problems often associated with the chemical reactions involved in casting include cure shrinkage,

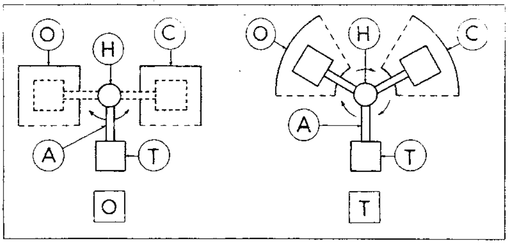

Fig. 21 Swing/rotary arm rotational molding: $\boxed{\text{O}}$, one-arm (swing); $\boxed{\text{T}}$, three-arm (rotary); O, oven; H, hub; C, cooling chamber; T, mold; A, arm. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

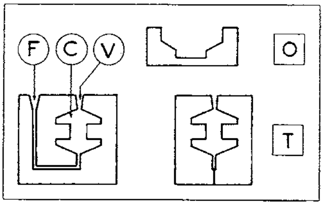

Fig. 22 Casting molds: $\boxed{\text{O}}$, open mold; $\boxed{\text{T}}$, two-piece mold; F, feed runner; C, cavity; V, vent. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

which can be as high as about 20% for acrylics, and heat evolution (exothermic reactions), which can lead to runaway situations through autoacceleration.

11 COMPRESSION MOLDING

Principle

The compression-molding process is used almost exclusively for temperature-activated thermosetting polymers. Compression molding basically involves the pressing of a deformable material between the two halves of a heated mold, as shown in Fig. 23, and its transformation into a solid product under the effect of the elevated mold temperature. Compression molding temperatures are often in the range of 140–200°C; mold pressures can vary from about 35 atm (~500 psi) to 700 atm (~10,000 psi); and setting (curing, vulcanization) times can vary widely from about 1 min for relatively thin phenolic parts to over 1 h for very thick rubber components. Material charges are often preheated or preplasticated to speed up the initial softening stage.

Equipment

Compression-molding molds are used on compression presses that may be of the downstroking $\boxed{\text{D}}$ or upstroking $\boxed{\text{U}}$ types, and are generally slow-acting and hydraulically operated (clamping ram or cylinder).

Tooling

Compression molds for thermosetting resins and vulcanizable rubbers are heated to temperatures typically in the range of 140–200°C (~280–400°F) and are

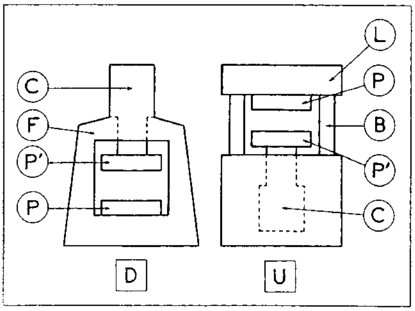

Fig. 23 Compression-molding presses: $\boxed{\text{D}}$, downstroking; $\boxed{\text{U}}$, upstroking; P, stationary platen; P′, moving platen; F, frame; C, clamping cylinder; L, bolster plate; B, tie bar. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

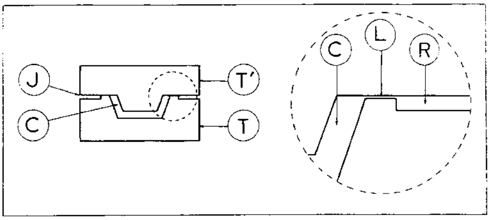

Fig. 24 Flash-compression mold: C, cavity; J, parting line; T, T′, mold halve; L, flash land; R, flash recess. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

subjected to high forces and pressures. In compression molding, it is the closing of the two-half mold that shapes the material charge and establishes the pressure required to obtain a good molded part. Flash molds are illustrated in Figs. 24 and 25.

Auxiliaries

The starting material for compression molding is often in powder form for thermosetting systems such as phenolic resins and amino resins. The commercial molding of small parts calls for the use of multiple cavity molds. Material charges are generally preheated to temperatures somewhat below those initiating rapid curing.

Materials

Temperature-activated thermosetting plastics most commonly processed by compression molding include phenolic, amino and alkyd resins, and diallyl phthalate (DAP), as well as unsaturated polyesters (UP) and epoxy resins (EP). They are often associated with particulate fillers and fiber reinforcement.

12 TRANSFER MOLDING

Principle

In transfer molding, a softened temperature-activated thermoset, or a vulcanizable rubber, is transferred through a narrow gate into the closed cavity of a heated mold, where it cures to a solid state.

Equipment

Hydraulic presses similar to compression-molding presses are used for transfer molding.

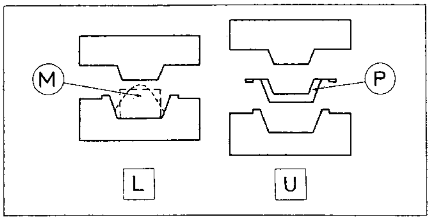

Fig. 25 Flash molding: $\boxed{\text{L}}$, loading; $\boxed{\text{U}}$, unloading; M, material charge; P, product. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

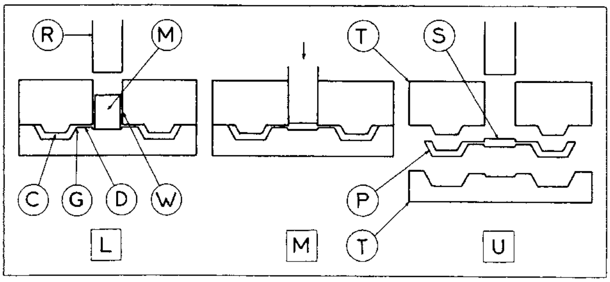

Fig. 26 Plunger transfer molding: $\boxed{L}$, loading; $\boxed{M}$, molding; $\boxed{U}$, unloading; W, loading well; D, distributing channel; G, gate; C, cavity; R, plunger; M, material charge; T′, stationary mold half; S, cull (scrap); P, product; T, moving mold half. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

Tooling

There are two basic methods for transfer molding. These are *plunger transfer molding* and *pot transfer molding*. In plunger transfer molding (Fig. 26), the material charge is first placed in a loading well, which extends to the mold parting line $\boxed{L}$. The plunger then transfers the material to the cavities, through distributing channels and gates $\boxed{M}$. The material left at the bottom of the loading well, known as the "transfer pad" (cushion, cull), is essential for pressure control but should be minimal. Products and a certain amount of material, to be scrapped, are ejected after mold opening $\boxed{U}$.

In pot transfer molding (Fig. 27), the material charge is loaded into the pot (chamber), and subsequently transferred through the sprue and gate into the cavity. The press stroke can normally assure both old clamping and material transfer functions, without a need for an auxiliary transfer ram.

Materials and Products

Transfer molding is normally used with materials that have fairly high precuring fluidity, facilitating the flow from the loading areas to mold cavities.

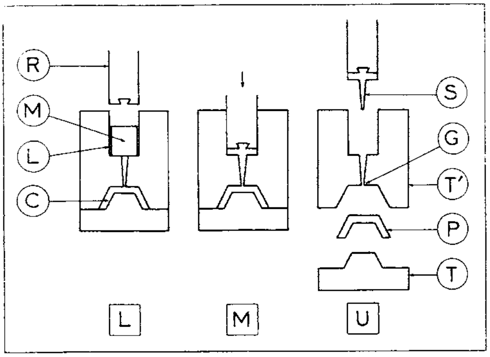

Fig. 27 Pot transfer molding: $\boxed{L}$, loading; $\boxed{M}$, molding; $\boxed{U}$, unloading; C, cavity; L, pot; M, material charge; R, transfer piston; S, sprue (scrap); G, gate; T, T′, mold halves; P, product. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

13 INJECTION MOLDING

The injection-molding process involves the rapid pressure filling of a shape-specific mold cavity with a fluid material, followed by the solidification of the material into a product. The process is used for thermoplastics, thermosetting resins, and rubbers.

Principle

The injection molding of thermoplastics can be subdivided into several stages, as illustrated in Fig. 28. At the plastication stage $\boxed{\text{P}}$, the feed unit $\boxed{\text{F}}$ operates in much the same way as an extruder, melting and homogenizing the material in the screw/barrel system. The screw, however, is allowed to retract, to make room for the molten material in a space at the cylinder head, referred to here as the material "reservoir," between the screw tip and a closed valve or an obstruction of solidified material from the previous shot. At the injection stage $\boxed{\text{I}}$, the screw is used as a ram (piston) for the rapid transfer of the molten material from the reservoir to the cavity between the two halves (T and T′) of the closed mold. Since the mold is kept at a temperature below the solidification temperature of the material, it is essential to inject the molten material rapidly to ensure complete filling of the cavity. A high holding or packing pressure (10,000–30,000 psi or 600–2000 atm) is normally exerted, to partially compensate for the thermal contraction (shrinkage) of the material upon cooling. The cooling of the material in the mold is often the limiting time factor in injection molding because of the low thermal conductivity of polymers. After the cooling stage, the mold can be opened and the solid product removed (ejection stage $\boxed{\text{E}}$).

Equipment

Injection molding machines are now most commonly of the reciprocating screw type, as illustrated in Fig. 29. Two distinct units referred to as the feed unit $\boxed{\text{F}}$

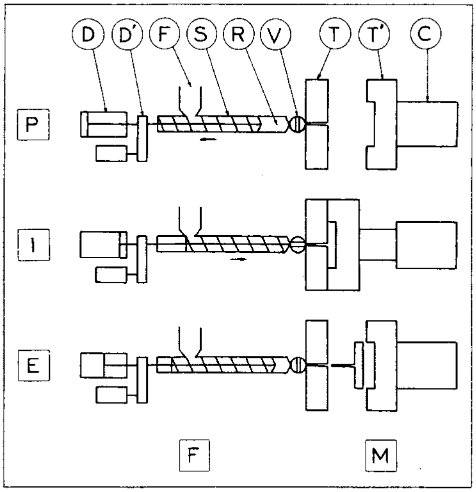

Fig. 28 Injection-molding principle: $\boxed{\text{F}}$, feed unit; $\boxed{\text{M}}$, mold unit; $\boxed{\text{P}}$, plastication stage; $\boxed{\text{I}}$, injection;; $\boxed{\text{E}}$, product ejection (demolding); D, axial screw drive; D′, rotation screw drive; F, feed hopper; S, reciprocating screw; R, material "reservoir"; V, valve; T, T′, mold halves; C, mold clamping device. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

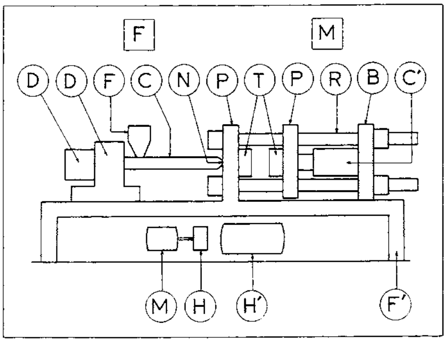

Fig. 29 Injection-molding machine: $\boxed{\text{F}}$, feed unit; $\boxed{\text{M}}$, mold unit; D, axial screw drive; D′, rotation screw drive; F, feed hopper; C, plastication/injection cylinder; P, stationary platen; T, mold; P′, moving platen; R, tie bars; B, back platen; C′, mold clamping mechanism; F′, maching grame; H′, hydraulic accumulator; H, hydraulic pump; M, electric motor; In, injection unit. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

and the mold unit $\boxed{\text{M}}$, are mounted on a frame (F′). The feed unit $\boxed{\text{F}}$ consists of the plastication/injection cylinder (screw, barrel, and feed hopper), the axial screw drive, and the rotation screw drive.

While injection-molding machines may occasionally be dedicated to the molding of a single product, a machine is normally used with a variety of tools (molds), which may imply frequent mold changes and the associated costly setup period. Injection-molding machines are available in a broad range of sizes. They are normally rated by their maximum clamping force, with normal ranges from about 25–150 tons for "small" machines, 150–750 tons for "medium-size" machines, and 750–5000 tons for "large" machines; the current maximum is 10,000 tons. Figure 30 shows several common mold-clamping mechanisms employed in the injection-molding process.

Tooling

The interchangeable injection-molding tool, the *mold*, must (a) provide a cavity corresponding to the geometry of the product and (b) allow the ejection of the

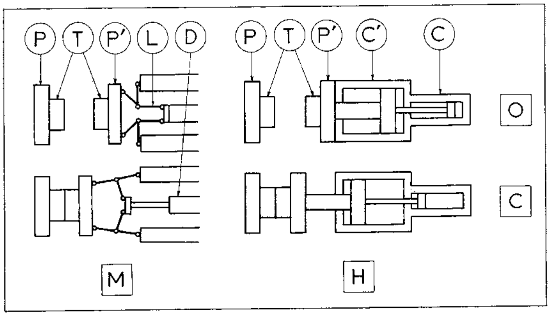

Fig. 30 Mold-clamping mechanisms: $\boxed{\text{M}}$, mechanical (double toggle); $\boxed{\text{H}}$, hydraulic (dual cylinder); $\boxed{\text{O}}$, mold open configuration; $\boxed{\text{C}}$, mold closed configuration; P, stationary platen; T, mold; P′, moving platen; L, link, D, toggle drive; C′, lock cylinder; C, traverse cylinder. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

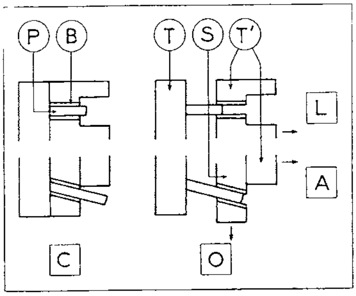

Fig. 31 Pin/busing: $\boxed{\text{L}}$, centering pin; $\boxed{\text{A}}$, angled pin; $\boxed{\text{C}}$, mold closed configuration; $\boxed{\text{O}}$, mold open configuration; P, pin; B, busing; T, stationary mold half; S, secondary mold section; T′, moving mold half. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

product after its solidification. Primary mold opening is achieved by fastening one-half of the mold to the stationary platen (T), as shown in Fig. 31, and the other half to the moving platen (T′). The stationary mold half is sometimes referred to as the "front," "cavity," or "negative block," and the moving mold half as the "rear," "force," or "positive block." The removal of a product from a cavity surface requires, in addition to an ejection system, a suitable surface finish and an appropriate taper or draft. It need not require a mold release agent.

During injection, the material flows from the nozzle at the tip of the injection unit to the single cavity, or to each of several cavities, through what is referred to here as the "feed system," generally comprising sprues, runners, and gates, as illustrated in Fig. 32. In most cases, injection-molded products need to be removed from one mold half by an ejection (knockout, stripping) device. This device is normally incorporated in the moving mold half. Retractable secondary mold sections may be required when products feature undercuts, reentrant shapes, internal or external threads, etc.

Runners are machined in mold halves, next to the parting surface. One solution, applicable to chemically stable thermoplastics, consists of having large runners cooled in such a way that a sleeve of insulating solid plastic forms around a molten core, where the intermittent injection flow takes place; this method is referred to as "insulated" or "Canadian" runner molding. Another solution referred to as "hot runner molding" involves a heated runner, or man-

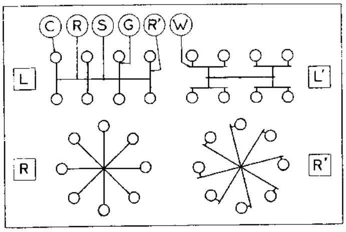

Fig. 32 Multicavity molding: $\boxed{\text{L}}$, $\boxed{\text{L}}$′, linear arrangements; $\boxed{\text{R}}$, $\boxed{\text{R}}$′, radial arrangements; C, cavity; R, main runner; S, sprue; G, gate; R′, secondary runner; W, cold slug well. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

ifold block, and is often used in conjunction with valve gating. Gates serve several purposes in injection molding. Their easily altered, smaller cross section permits a convenient control of the flow of the molten material, the rapid freezing of the material to shut off the cavity after injection, and the easy separation of the products from the feed appendage (degating). Important savings can be made by using hot runners.

The maximum pressure in injection molds is normally in the range of 4000–12,000 psi corresponding to a clamping force per unit projected area of cavity and feed system in the range of 2–6 tons/in.^{2}. The construction of injection molds requires materials with a combination of good thermal conductivity and resistance to mechanical wear and abrasion. Prototype molds can be cast from low melting alloys. Short-run molds (about 1000 moldings) can be machined in aluminum or mild steel. For medium runs (about 10,000–100,000 moldings), tool steel is normally used. Long runs involving millions of moldings require special hardened and chrome-plated steels.

A variety of techniques are used to form mold cavities: cutting (machining) of a solid block, computer-aided machining (CAM) centers, hobbing (cold forming), electrochemical machining (ECM), electrical discharge machining (EDM), or spark erosion, electroforming, plating, and etching.

Auxiliaries

Many thermoplastic resins require thorough drying prior to molding, to avoid the formation of voids or a degradation of the material at molding temperatures. Mold temperature control is often achieved by the circulation of a fluid through a separate heater/chiller device. With increased interest in automation, robots have been introduced for the removal of products and feed appendages from open molds, and for separation (degating) and sorting. Feed appendages, startup scrap, and occasional production scrap are normally reground in granulators and recycled as a fraction of the feed material.

Materials

All thermoplastics are, in principle, suitable for injection molding, but since fast flowrates are needed, grades with good fluidity (high melt index) are normally preferable.

Products

A major advantage of injection-molded products is the incorporation of fine details such as bosses, locating pins, mounting holes, bushings, ribs, flanges, etc., which normally eliminates assembly and finishing operations. Thermosetting resin systems, such as phenolics (PF) or unsaturated polyester (UP), often used with fillers or reinforcements, are increasingly injection molded at relatively high speeds. Curing, which involves chemical reactions, takes generally much longer than the injection, and multimold machines are thus often used with shuttle or rotary systems. Injection molding is increasingly used for producing relatively small rubber products significantly faster than by compression molding and, normally, with a smaller amount of scrap and a better dimensional accuracy. As in the case of thermosetting resins, a heated mold is needed for vulcanization (curing).

14 REACTION INJECTION MOLDING

Principle

Reaction (or reactive) injection molding (RIM) is a relatively new process that involves the rapid mixing, in precise proportions, of two or more highly reactive low-molecular-weight liquid components, and the immediate injection of the mixture into a closed mold. Polymerization and/or molecular network formation take place in the mold in a very short time. The RIM process was developed with urethane resins and is still used primarily with these materials.

Equipment

RIM process equipment involves two, and sometimes more, components, each one corresponding to a stream up to the mixing head, as illustrated in Fig. 33. A suitable batch of each component is held in a material tank (day tank, work tank), which is pressure-rated (about 7.5 atm or ~110 psi). Tanks generally feature a nitrogen gas overlay (blanket) to protect the chemicals from oxygen or moisture, and a vortex-free stirrer (agitator), particularly for materials containing filler particles. A heat exchanger permits the control of the temperature of each component. Some resin systems (polyamide systems in particular) require much higher processing temperatures; corresponding equipment featuring jacketed tanks and lines is often referred to as "heat-traced" (HT) equipment.

Tooling

RIM molds do not have to withstand high pressures (4–8 atm or ~50–100 psi), and for most polyurethane systems their temperature remains moderate. Depending on the number of parts required, mold materials can range from epoxy, metal-coated epoxy (nickel is common), kirksite, aluminum, and steel.

Materials

Urethane resin systems (RIM-PUR) are most widely used. Urea resin systems (RIM-PUA) have been developed with a superior temperature resistance that makes them suitable, for example, for automotive body components subjected to high-temperature paint ovens.

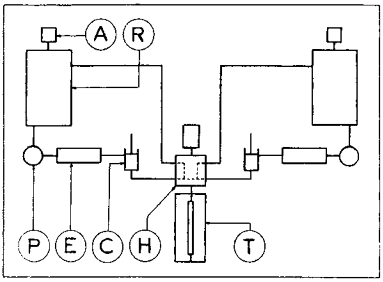

Fig. 33 RIM equipment: T, mold; H, mixing head; E, heat exchanger; R, recirculating pump; R, material tank; A, stirrer; C, metering cylinder. (From Charrier, J.-M., *Polymeric Materials and Processing: Plastics, Elastomers and Composites,* Hanser Publishers, Munich, 1990.)

Products

Because of the low-viscosity fluid involved and the low mold pressure, the RIM process is particularly well suited to the fabrication of large thin parts, which include exterior automotive body panels such as front-end fascias and fenders, as well as furniture, electronic cabinets, and window frames.

15 SUMMARY AND CONCLUSIONS

This chapter has highlighted the most useful of the plastics parts processing techniques. It has attempted to elucidate the fundamental processing principle underlying each technique, describe the equipment and tooling employed to carry out this processing technique, discuss the materials most commonly used with each processing technique, and point out the most important products manufactured using that technique.

To gain a more complete understanding of plastics parts processing, one should refer to the core literature on the subject as listed in the following section.

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CHAPTER 33
PLASTIC PARTS PROCESSING II

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1 CLASSIFICATION OF PLASTIC PARTS PROCESSES

Plastic parts processes may be classified by the time mode of product formation and subclassified by the dominating kinematics of the associated flows. Sometimes *dimensionality* is used, referring to a product as “one,” “two,” or “three dimensional.”

Handbook of Materials Selection. Edited by Myer Kutz

1.1 Continuous and Cyclic Processes

The two time mode categories of processing are *continuous* and *cyclic*. Products made continuously are characterized by having one dimension that is "unending." Consequently, the other two dimensions constitute an area referred to as the "profile." The profile of a rod is a circle; the profile of a pipe is the area between two concentric circles. Of course, many useful profiles, such as door closures, are geometrically complicated; however, they share the characteristic of having the same cross section throughout their length.

These processes are also referred to as *steady state*. The design equations used to relate the dimensions of the profile to the plastic's properties and operating parameters do not include "time" as an independent variable.

Products that are produced cyclically are inherently three dimensional; that is, no one dimension is unending—each is finite. Every part, though replicated cycle after cycle, has its own identity.

Such processes are also referred to as *unsteady state*. There is a start and end to each cycle, the *cycle time*. And, the design equations always include time as an *independent* variable; however, in terms of the process economics, cycle time is an important *dependent* variable.

1.2 Dominating Kinematics of Flow*

Kinematics is the study of motion, per se, without regard to the forces necessary to produce that motion. In plastics parts processing, there are two such categories—shear flow and extensional flow. Although these flows are usually characterized in rectangular, cylindrical, bipolar, or some other appropriate three-dimensional coordinate system, it is convenient to use a generic "numerical" representation to describe them. Let the fluid's velocity **v**, be decomposed into its three components, (v_1,v_2,v_3), in the three coordinate directions, $N(x_1,x_2,x_3)$, where N is used to designate the numerical coordinate system.

Shear-Flow-Dominated Processes

Shear flows are those motions for which $(i, j = 1, 2, 3)$

$$v_i = f(x_j) \qquad i \neq j \tag{1.1}$$

The shear rate $\dot{\gamma}_{ji}$ is given by

$$\dot{\gamma}_{ji} \equiv \frac{\partial v_i}{\partial x_j} \tag{1.2}$$

Simple shear has the universally accepted description

$$\mathbf{v} = (v_1,0,0) = N(0,x_2,0) \tag{1.3}$$

There is only one nonzero component of motion, and it is a function of only

---

*Note: Scalars are *italic,* vectors are **boldface,** and tensors are ***boldface italic***.

one other, and different, direction. More complicated flows may be characterized as *complex shear*, when there are two nonzero components of the velocity, or *total shear*, when all three components are nonzero, provided in each case that Eq. 1.1 is satisfied for each i and j.

Extrusion is a shear-dominated continuous process and injection molding is a shear-dominated cyclic process.

Extensional-Flow-Dominated Processes

Extensional flows are those motions for which

$$v_i = f(x_i) \tag{1.4}$$

i.e., Eq. 1.1 with $i = j$. The extensional rate is

$$\dot{\varepsilon}_{ii} \equiv \frac{\partial v_i}{\partial x_i} \tag{1.5}$$

There are three such extensional flows: uniaxial, planar, and uniform biaxial. Uniaxial extensional flow has the characteristics

$$\mathbf{v} = (v_1,v_2,v_3) = N(x_1,x_2,x_3) \tag{1.6}$$

where $\partial v_1/\partial x_1 > 0$ and $\partial v_2/\partial x_2 = \partial v_3/\partial x_3 = -\frac{1}{2}(\partial v_1/\partial x_1)$. It is uniaxial because only one component of velocity is increasing with position; the other two are decreasing at rates such that the volume remains constant. Fiber spinning is a continuous uniaxial-dominated extensional process.

Planar extensional flow has the characteristics

$$\mathbf{v} = (v_1,v_2,0) = N(x_1,x_2,x_3) \tag{1.7}$$

where $\partial v_1/\partial x_1 > 0$ and $\partial v_2/\partial x_2 = -(\partial v_1/\partial x_1)$ and $\partial v_3/\partial x_3 = 0$ since $v_3 = 0$. It is planar because only two components of velocity change; one is increasing with position and another is decreasing at a rate such that the volume remains constant. The third "does not change" with position because that component is zero. Sheet extrusion, with pulling to reduce thickness, is a continuous planar-dominated extensional process.

Uniform biaxial extensional flow has the characteristics

$$\mathbf{v} = (v_1,v_2,v_3) = N(x_1,x_2,x_3) \tag{1.8}$$

where $\partial v_1/\partial x_1 = \partial v_2/\partial x_2 > 0$ and $\partial v_3/\partial x_3 = -2(\partial v_1/\partial x_1)$. It is uniform biaxial because two components of velocity increase with position at the same rate; the third decreases at a rate such that the volume remains constant. Film blowing is a continuous process dominated by uniform biaxial extension.

Quiescent Process

A plastic parts process that has no flow is casting; it is a quiescent process and is inherently cyclic.

1.3 Sources of Momentum

Since force **F** equals mass *m* times acceleration **a,** and acceleration is the time rate of change of velocity **v**, and momentum is mass times velocity, then (for constant mass)

$$\mathbf{F} = m\,\mathbf{a} = \frac{m\,d\mathbf{v}}{dt} = \frac{d(m\mathbf{v})}{dt}$$

This force per unit area A, which is a stress $\boldsymbol{\tau}$, that produces a shear or extensional motion is a flux of momentum, the time rate of momentum per unit area.

The magnitudes and directions of the fluxes of momentum are given by the stress tensor $\boldsymbol{\tau}$. In component form the first subscript designates the direction in which the momentum is being transferred, and the second designates the direction in which the fluid is moving (the velocity).

$$\boldsymbol{\tau} \equiv \begin{bmatrix} \tau_{11} & \tau_{12} & \tau_{13} \\ \tau_{21} & \tau_{22} & \tau_{23} \\ \tau_{31} & \tau_{32} & \tau_{33} \end{bmatrix} = \begin{bmatrix} \tau_{11} & \tau_{12} & \tau_{13} \\ \tau_{12} & \tau_{22} & \tau_{23} \\ \tau_{13} & \tau_{23} & \tau_{33} \end{bmatrix} \tag{1.9}$$

The six different components of this three-by-three symmetric matrix characterize the rheological properties of a polymer melt.

In plastic parts processing, the sources of momentum may be classified as "drag," "pressure," "gravitational," or "centrifugal"; hence, the processes may be given those names.

Drag-Flow Processes

Drag-flow processes are those in which a moving object, usually metal, supplies the momentum that is transferred to the plastic, causing it to move. In extrusion it is the turning screw; in wire coating it is the moving wire; in calendering it is the rotating rolls.

Pressure-Flow Processes

Pressure may be a *source* of momentum that produces the flow, a *source* of momentum that opposes the flow, or a *sink* of momentum reducing the magnitude of the flow. Pressure is a "positive" source when an extrudate flows through a die; pressure is a "negative" source for the melt within the channels of an extrusion screw; the injection pressure in a molding process is required because there is a pressure drop as the melt flows through the sprue, runner, gate, and cavity.

Vacuum, a negative pressure, is an essential operating parameter in thermoforming; it is the agent that removes the air between the sheet and the mold, and the resulting positive pressure gradient across the sheet causes the sheet to take on the shape of that mold.

Gravitational-Influenced Processes

Some processes, such as vertical fiber spinning, benefit from gravity as a source of momentum, which helps to reduce the diameter of the fiber. Gravity aids the sheet movement in thermoforming, if the mold is below the plastic.

Centrifugal-Based Process

Rotational molding relies on the centrifugal forces developed from two rotating axes to press the plastic melt against the inside surfaces of the mold.

2 INTRODUCTION

Engineering problems are usually one of two types: If a machine needs to be specified for a given task, the problem is one of design; if the performance of an existing machine needs to be characterized, the problem is one of rating. In the former situation, there are usually more variables to be specified than there are equations; constraints must be assigned. In the latter situation, the constraints are fixed; they limit the variation in performance that may be achieved through changes in the operating parameters.

2.1 Design Problem

In the design problem, some measure of performance must exist and be known. This constitutes the dependent variable. It is a function of three categories of variables—the geometric dimensions of the shaping apparatus, the properties of the plastic, and the operating parameters.

Measure of Performance

In continuous processes, this is usually the rate of production of a given profile. In extrusion, this is the rate of production of a specified shape; in wire coating, it is the rate of production of a wire with a specified coating thickness; in calendering, it is the rate of production of a sheet of specified width and thickness.

In cyclic processes, the measure of performance is usually the number of parts produced annually, monthly, weekly, or hourly, which is related to the reciprocal of the cycle time. A component of the cycle time is the processing time—the time it takes to shape the object; other components of the cycle time may be the time required to load a preform and unload the product, close and open a mold, and/or solidify the plastic.

Geometric Factors

The objects whose geometric dimensions may influence the performance of a process include the barrel, screw, and die in extrusion; the wire and die in wire coating; the rolls in calendering; etc. In general, these characteristics cannot be changed during processing. However, there are other distances that can be changed, such as the gap between the two plates that constitutes a lip die or the movable core that determines the thickness of the parison in blow molding.

Material Properties

Although the rheological properties of the polymer melt (defined in Section 2.2) determine its motion, other physical and thermal properties have an influence on the measure of performance. These include the density ρ, the heat capacity C_P, and the thermal conductivity k.

Operating Parameters

An operating parameter of a process might be the translational or rotational speed of a moving object (such as the wire in wire coating, the screw in extrusion, or the roll in calendering). Another such parameter is the pressure in injection molding, blow molding, or film blowing.

2.2 Design Solution Algorithm

The following procedure, or algorithm, gives one a logical approach for obtaining a solution to each processing problem.

- One should draw a picture of the process, identifying an appropriate geometry and the associated dimensions.
- Identify the nonzero components of the velocity and the spatial coordinates of which they are functions.
- Write and simplify the equation of continuity.
- Write and simplify the equation(s) of motion.
- If variations in temperature are to be included, write and simplify the equation of energy.
- Combine the above solutions or solve those equations simultaneously to determine an expression for the measure of performance.

Geometry of Process

Plastic parts processes may be described by using rectangular, cylindrical, bipolar, or spherical geometries. In a rectangular coordinate system, the standard order is $R(x,y,z)$. In a cylindrical coordinate system, the standard order is $C(r,\theta,z)$, where $0 < \theta < 2\pi$. The bipolar system is two nonconcentric cylindrical systems with parallel axes; it is particularly useful in describing the wire coating process to make household wiring. In a spherical coordinate system, the standard order is $S(r,\theta,\phi)$, where $0 < \theta < \pi$ and $0 < \phi < 2\pi$; these angles correspond to latitudes and longitudes of Earth, respectively (although the latter are measured $\pm 90°$ north and south and $\pm 180°$ east and west).

Velocity Flow Field

Having specified an appropriate geometric coordinate system, one must specify the directions in which the velocity components are nonzero. If possible, one also identifies the directional dependencies of these components. These decisions help to define the process kinematics.

Equation of Continuity

The equation of continuity (EOC) is the differential form of the conservation of matter. Polymer melts are regarded as being incompressible; this is true except for injection molding where high pressures are involved. In vector form, the EOC is

$$-\mathrm{div}(\rho \mathbf{v}) = \frac{\partial \rho}{\partial t} \tag{2.1}$$

for the unsteady-state flow of compressible fluids,

$$\mathrm{div}(\rho \mathbf{v}) = 0 \tag{2.2}$$

for the steady-state flow of compressible fluids, and

$$\mathrm{div}(\mathbf{v}) = 0 \tag{2.3}$$

for the flow of incompressible fluids, whether unsteady or steady state. [*Note:* the vector calculus operators divergence (div) and gradient (grad) permit a concise presentation of equations; the complete component forms in several coordinate systems are readily available in a number of texts, including those by Bird et al.[1,2]]

Kinematics of Flow

The kinematics of the processing flows should be identified because these determine which rheological properties of the polymer melt will influence the measure of performance.

Rheological Properties of Polymer Melt

There are six rheological properties of a polymer melt that influence the measures of performance. If the processing flow is dominated by shear, then (i) the shear viscosity η and the (ii) first Ψ_1 and (iii) second Ψ_2 normal stress functions of the melt will play important roles in determining the shape of the plastic part. These properties are defined as follows:

$$\eta \equiv -\frac{\tau_{21}}{\dot{\gamma}_{21}} \tag{2.4}$$

$$\Psi_1 \equiv -\frac{\tau_{11} - \tau_{22}}{(\dot{\gamma}_{21})^2} \tag{2.5}$$

$$\Psi_2 \equiv -\frac{\tau_{22} - \tau_{33}}{(\dot{\gamma}_{21})^2} \tag{2.6}$$

where

$$\boldsymbol{\tau} \equiv \begin{bmatrix} \tau_{11} & \tau_{12} & \tau_{13} \\ \tau_{12} & \tau_{22} & \tau_{23} \\ \tau_{13} & \tau_{23} & \tau_{33} \end{bmatrix} = \begin{bmatrix} \tau_{11} & \tau_{12} & 0 \\ \tau_{12} & \tau_{22} & 0 \\ 0 & 0 & \tau_{33} \end{bmatrix} \tag{2.7}$$

It is known from experimental data that $\Psi_1 > 0$, $\Psi_2 < 0$, and $|\Psi_1| > |\Psi_2|$.

If the processing flow is dominated by an extensional flow, then either (i) the uniaxial extensional viscosity η_3, (ii) the planar extensional viscosity η_4, or (iii) the uniform biaxial extensional viscosity η_6 will play an important role in determining the size of the product. (The numerical subscripts are those originally used by Denson.[3])

These properties are defined as follows:

$$\eta_3 \equiv -\frac{\tau_{11} - \tau_{22}}{\dot{\varepsilon}_{11}} \tag{2.8}$$

$$\eta_4 \equiv -\frac{\tau_{11} - \tau_{22}}{\dot{\varepsilon}_{11}} \tag{2.9}$$

$$\eta_6 \equiv -\frac{\tau_{11} - \tau_{33}}{\dot{\varepsilon}_{11}} \tag{2.10}$$

where the constraints on $\dot{\varepsilon}_{ii}$ are as given in Section 1.2 and

$$\boldsymbol{\tau} \equiv \begin{bmatrix} \tau_{11} & \tau_{12} & \tau_{13} \\ \tau_{12} & \tau_{22} & \tau_{23} \\ \tau_{13} & \tau_{23} & \tau_{33} \end{bmatrix} = \begin{bmatrix} \tau_{11} & 0 & 0 \\ 0 & \tau_{22} & 0 \\ 0 & 0 & \tau_{33} \end{bmatrix} \tag{2.11}$$

Constitutive Equations for Polymer Melt

The simplest model that relates τ_{21} and $\dot{\gamma}_{21}$ is that for a Newtonian fluid, i.e., Newton's law:

$$\mu = -\frac{\tau_{21}}{\dot{\gamma}_{21}} \neq f(\dot{\gamma}_{21}) \tag{2.12}$$

i.e., μ is a constant. If the fluid requires τ_{21} to exceed an initial yield value, τ_0, after which Newton's law is applicable, the material is known as a Bingham plastic[4]:

$$\mu_B = -\frac{\tau_{21} - \tau_0}{\dot{\gamma}_{21}} \neq f(\dot{\gamma}_{21}) \tag{2.13}$$

Although there have been a myriad of equations presented in the literature (e.g., as cited by Skelland[5]) to describe the shear viscosity of a non-Newtonian fluid, almost all may be reduced to one of the three following generalized forms:

Generalized Eyring Model. A generalized Eyring model may be written as

$$\Gamma \equiv \frac{\eta - \eta_\infty}{\eta_0 - \eta_\infty} = \frac{\text{arcsinh}(\dot{\gamma}/\dot{\gamma}_a)}{\dot{\gamma}/\dot{\gamma}_a} \tag{2.14}$$

where $\dot{\gamma}_a$ is the shear rate at which the viscosity is η_a and $\Gamma_a \equiv (\eta_a - \eta_\infty)/(\eta_0 - \eta_\infty) = \text{arcsinh}(1) = 0.8814$. Since η_0 and η_∞ are properties of the liquid, this model has no adjustable parameters.

With $\eta_\infty = 0$, this has been called the Sutterby model[6] (in which the $\dot{\gamma}_a$ was thought to be arbitrary) or the Eyring-Prandtl model.[7,8] When $\eta_\infty \neq 0$, it is called the Eyring-Powell model,[9] which is sometimes shown in a pseudo-two-parameter form. O'Nan[10] investigated, among other models, one from his own thought (a shear stress version of the Eyring-Powell model):

$$\Gamma \equiv \frac{\eta - \eta_\infty}{\eta_0 - \eta_\infty} = \frac{\operatorname{arcsinh}(\tau/\tau_a)}{\tau/\tau_a} \tag{2.15}$$

where τ_a is the shear stress at which the viscosity is η_a and $\Gamma_a \equiv (\eta_a - \eta_\infty)/(\eta_0 - \eta_\infty) = \operatorname{arcsinh}(1) = 0.8814$; it has no adjustable parameters.

Generalized Yasuda Model. A generalized Yasuda model may be written as

$$\Gamma \equiv \frac{\eta - \eta_\infty}{\eta_0 - \eta_\infty} = \frac{1}{[C + (\dot\gamma/\dot\gamma_a)^m]^{(1-n)/m}} \tag{2.16}$$

This model gives the viscosity as a function of shear rate; C must be unity for the equation to satisfy the low-shear-rate limit criterion. However, when $C = 0$ and $\eta_\infty = 0$, this model becomes the power law model, credited to Ostwald[11] and de Waele.[12] This is often used, because of its simplicity, as the lowest-level non-Newtonian model in polymer processing calculations; it is inherently incorrect because it predicts an infinite viscosity wherever the shear rate is zero. (In polymer processing, the shear rate is zero whenever a geometric symmetry exists, such as along the centerline of a circular conduit or in the center plane between two parallel plates; it is not a trivial situation that may be ignored.) If $C = 0$ and the power law range is truncated at η_0, then one has the Spriggs model.[13] If $C = 0$ and $\eta_0 - \eta_\infty = K$, then one has the Sisko model.[14]

With $C = 1$, the slope of the high shear-rate range of the $\ln \Gamma$ vs. $\ln \dot\gamma$, or $\ln(\eta - \eta_\infty)$ vs. $\ln \dot\gamma$, curve is $-(1 - n)$, from which n may be determined. [Note that since n is a positive number less than unity, $-(1 - n)$ cannot be more negative than -1.] $\dot\gamma_a$ is the shear rate at an arbitrary data point through which one wishes the equation to pass; m is determined from the equation

$$m = \frac{(1 - n)(\ln 2)}{\ln(1/\Gamma_a)} \tag{2.17}$$

where $\Gamma_a = (\eta_a - \eta_\infty)/(\eta_0 - \eta_\infty)$ and η_a is the viscosity at $\dot\gamma_a$. If η_∞ is not known, then the $\ln(\eta)$ vs. $\ln(\dot\gamma)$ curve is used to determine n, and η_0/η_a replaces $1/\Gamma_a$ in the equation for m. In this case, a convenient arbitrary data point is the "corner shear rate," $\dot\gamma_c$, defined as the value of $\dot\gamma$ at which the η_0 line intersects the extrapolation of the high shear-rate line. At this shear rate, the corresponding value of the viscosity is η_c, and $m = (1 - n)(\ln 2)/\ln(\eta_0/\eta_c)$.

Since m is determined by n and the arbitrary point selected, this is a "*two* adjustable-parameter" model—the Yasuda model.[15] If $m = 2$, it is the Carreau model[16] that passes through the "no longer arbitrary" point $(\eta_a, \dot\gamma_a)$, where $\Gamma_a = \frac{1}{2}^{(1-n)/2}$. For $m = 1$, with $\eta_\infty = 0$, it is the Cross model[17] that passes through the point where $\eta_a = \eta_0/2^{(1-n)}$. These two models have one adjustable parameter, n. The Cross model is utilized in "C-MOLD," a software package for polymer processing. (C-MOLD is a registered trademark of Mold-Flow, Louisville KY).

Generalized Meter Model. A generalized Meter model may be written as

$$\Gamma \equiv \frac{\eta - \eta_\infty}{\eta_0 - \eta_\infty} = \frac{1}{[C + (\tau/\tau_a)^m]^{(1-n)/m}} \tag{2.18}$$

This model gives the viscosity as a function of shear stress. C must be unity for the equation to satisfy the low-shear-stress limit criterion. However, when $C = 0$ and $\eta_\infty = 0$, it yields the power law model.

With $C = 1$, the slope of the high-shear-stress range of the ln Γ vs. ln τ, or $\ln(\eta - \eta_\infty)$ vs. ln τ, curve is $-(1 - n)$, from which n may be determined. [Note that since n is a positive number less than unity, $-(1 - n)$ cannot be more negative than -1.] τ_a is the shear stress at an arbitrary data point through which one wishes the equation to pass; m is determined from the equation

$$m = \frac{(1 - n)(\ln 2)}{\ln(1/\Gamma_a)} \tag{2.19}$$

where $\Gamma_a = (\eta_a - \eta_\infty)/(\eta_0 - \eta_\infty)$ and η_a is the viscosity at τ_a.

If η_∞ is not known, then the ln η vs. ln τ curve is used to determine n, and η_0/η_a replaces $1/\Gamma_a$ in the equation for m. In this case, a convenient arbitrary data point is the corner shear stress, τ_c, defined as the value of τ at which the η_0 line intersects the extrapolation of the high-shear-stress line. At this shear stress, the corresponding value of the viscosity is η_c, and $m = (1 - n)(\ln 2)/\ln(\eta_0/\eta_c)$.

Sometimes this model is written as

$$\Gamma \equiv \frac{\eta - \eta_\infty}{\eta_0 - \eta_\infty} = \frac{1}{C + (\tau/\tau_a)^{\alpha-1}} \tag{2.20}$$

where C must be unity for the equation to satisfy the low-shear-stress limit criterion. Here, τ_a is the value of τ for which $\Gamma = \frac{1}{2}$, or where $\eta = (\eta_0 + \eta_\infty)/2$; hence, τ_a is frequently referred to as a mean value of τ. When $\eta_\infty \neq 0$ and $C = 1$, the equation is the Meter model.[18] When $\eta_\infty = 0$ and $C = 1$, it is the Ellis model.[19] When $\eta_\infty = 0$ and $C \neq 1$, it is the DeHaven model.[20] The first two are one-parameter models, and the third is an inappropriate two-parameter model.

If $C = 1$ and $\alpha = 3$, one has the Reiner-Philippoff model[21]; this model has no adjustable parameters.

Brodkey Model. The Brodkey model[22] is based on an analogy with the chemical kinetics of homogeneous reactions. It is especially versatile in that it can be used to describe time-dependent materials (using reaction rate constants) as well as time-independent ones (using reaction equilibrium constants); the other models included in this section are limited to the latter case. The time-dependent fluids are known as rheopectic, if the viscosity increases with the duration of shear, or thixotropic, if the viscosity decreases with the duration of shear.

Denny and Brodkey[23] expressed their model in terms of the fraction of molecules whose structure is unchanged by shear, F, according to the equation

$$F = \frac{f(\eta) - f(\eta_\infty)}{f(\eta_0) - f(\eta_\infty)} \tag{2.21}$$

where $f(\eta) = A\eta^{1/3.5}$. (Other researchers[24] would recommend 3.4, rather than 3.5.) Using U for "unchanged" and C for "changed," a kinetic equation for the reaction $U \leftrightarrow C$ may be written as

$$-\frac{dU}{dt} = k_1'U^m - k_2'C^n \quad \text{or} \quad -\frac{dF}{dt} = k_1'F^m - k_2'(1 - F)^n \tag{2.22}$$

in addition,

$$k_l' = k_l\tau_{ij}^{p1} \tag{2.23}$$

where $l = 1, 2$ and τ_{ij} is the shear stress. Combining these equations, one has

$$-\frac{dF}{dt} = k_1F^m\tau_{ij}^{p1} - k_2(1 - F)^n\tau_{ij}^{p2} \tag{2.24}$$

for time-dependent materials. For time-independent fluids, one sets $-dF/dt = 0$ to get

$$\frac{k_1}{k_2}\,\tau_{ij}^{p1-p2} = K\tau_{ij}^{p} = \frac{(1 - F)^n}{F^m} \tag{2.25}$$

where $K = k_1/k_2$ and $p = p_1 - p_2$. In these equations, the parameters are k_1, k_2, m, n, p_1, and p_2, or K, m, n, and p, respectively.

Rearranging this model into the normalized form used previously, we have $\eta = [f(\eta)/A]^{3.5}$; hence

$$\Gamma = \frac{f(\eta)^{3.5} - f(\eta_\infty)^{3.5}}{f(\eta_0)^{3.5} - f(\eta_\infty)^{3.5}} \tag{2.26a}$$

or more simply

$$\Gamma = \frac{f^{3.5} - f_\infty^{3.5}}{f_0^{3.5} - f_\infty^{3.5}} \tag{2.26b}$$

This can be expressed as

$$\Gamma = \frac{[(f_0 - f_\infty)F + f_\infty]^{3.5} - f_\infty^{3.5}}{f_0^{3.5} - f_\infty^{3.5}} \tag{2.27}$$

If $\eta_\infty = 0$, then $\Gamma = F^{3.5}$. Here F is the solution of $aF^b + F - 1 = 0$, where for time-independent materials

$$a = (K\tau_{ij}^p)^{1/n} \tag{2.28}$$

and for time-dependent materials

$$a = \left(\frac{k_1}{k_2}\,\tau_{ij}^{p1-p2}\right)^{1/n} \tag{2.29}$$

$$b = \frac{m}{n} \tag{2.30}$$

As explained by Brodkey,[22] m and n are small integers, as expected in chemical kinetics. If $m = n/2$, then

$$F = [-a + \sqrt{a^2 + 4}/2]^2 \tag{2.31a}$$

if $m = n$, then

$$F = \frac{1}{a + 1} \tag{2.31b}$$

if $m = 2n$, then

$$F = \frac{-1 + \sqrt{1 + 4a}}{2a} \tag{2.31c}$$

(The negative roots of these radicals are physically impossible.)

Corotational Jeffreys Model. The above equations say nothing about the normal stress functions. To do that one must use models based on either the corotational or codeformational time derivatives; the former is given by

$$\frac{\mathcal{D}\boldsymbol{T}}{\mathcal{D}t} = \frac{\partial \boldsymbol{T}}{\partial t} + \mathbf{v}\cdot\text{grad}\,\boldsymbol{T} + \frac{1}{2}\,(\boldsymbol{\omega}\cdot\boldsymbol{T} - \boldsymbol{T}\cdot\boldsymbol{\omega}) \tag{2.32}$$

where the tensor $\boldsymbol{T}$ may be either $\boldsymbol{\tau}$ or $\dot{\boldsymbol{\gamma}}$, each of which is symmetric.

The corotational Jeffreys model[25] expands the generalized Newtonian model by adding the corotational time derivatives of both the stress tensor and the rate of strain tensor. The adjustable parameters, λ_1 and λ_2, each have the units of time (i.e., seconds); hence, they may be thought of as stress and rate of strain time constants, respectively, of the melt:

$$\boldsymbol{\tau} + \lambda_1 \frac{\mathcal{D}\boldsymbol{\tau}}{\mathcal{D}t} = -\eta_0 \left(\dot{\boldsymbol{\gamma}} + \lambda_2 \frac{\mathcal{D}\dot{\boldsymbol{\gamma}}}{\mathcal{D}t} \right) \tag{2.33}$$

This model predicts the shear properties to be

$$\eta = \frac{\eta_0(1 + \lambda_1\lambda_2\dot{\gamma}_{21}^2)}{1 + \lambda_1^2\dot{\gamma}_{21}^2} \tag{2.34}$$

$$\Psi_1 = \frac{2\eta_0(\lambda_1 - \lambda_2)}{1 + \lambda_1^2\dot{\gamma}_{21}^2} \tag{2.35}$$

$$\Psi_2 = -\frac{\eta_0(\lambda_1 - \lambda_2)}{1 + \lambda_1^2\dot{\gamma}_{21}^2} \tag{2.36}$$

Note that, if $\lambda_1 > \lambda_2$, this model does predict that the first normal stress function (NSF) is positive and the second NSF is negative; it also predicts that $|\Psi_1| > |\Psi_2|$. However, the prediction that $-\Psi_2/\Psi_1 = 0.5$ is contrary to observations; experimental values of this ratio are not constant. This is a characteristic of each corotational model, and is one disadvantage of such a formulation.

This model predicts the extensional properties to be

$$\eta_3 = 3\eta_0 \tag{2.37}$$

$$\eta_4 = 4\eta_0 \tag{2.38}$$

$$\eta_6 = 6\eta_0 \tag{2.39}$$

These results reveal further deficiencies of this corotational model; it is no better than the Newtonian model for any extensional viscosity.

Criminale–Ericksen–Filbrey (CEF) Model. In conrast, the CEF model^{26} gives the absolutely correct expressions for the shear properties, yet defies utilitarian value!

$$\boldsymbol{\tau} = -\eta\dot{\boldsymbol{\gamma}} - \left(\frac{1}{2}\Psi_1 + \Psi_2\right)\dot{\boldsymbol{\gamma}}\cdot\dot{\boldsymbol{\gamma}} + \frac{1}{2}\Psi_1 \frac{\mathcal{D}\dot{\boldsymbol{\gamma}}}{\mathcal{D}t} \tag{2.40}$$

The results of the analysis for shear properties are

$$\eta = \eta \tag{2.41}$$

$$\Psi_1 = \Psi_1 \tag{2.42}$$

$$\Psi_2 = \Psi_2 \tag{2.43}$$

There are no parameters in the model to permit the curve fitting of experimental data. The identities simply demonstrate that the model is self-consistent; they cannot address the question of the model's validity.

For the extensional viscosities, the results (which are not trivial) are

$$\eta_3 = 3[\eta + (\tfrac{1}{2}\Psi_1 + \Psi_2)\dot{\gamma}_{21}] \tag{2.44}$$

$$\eta_4 = 4\eta \tag{2.45}$$

$$\eta_6 = 6[\eta - (\tfrac{1}{2}\Psi_1 + \Psi_2)\dot{\gamma}_{21}] \tag{2.46}$$

Although these equations correctly predict the Newtonian low-shear-rate limits of $3\eta_0$, $4\eta_0$, and $6\eta_0$, respectively, they are not explicit in the rate of strain, $\dot{\varepsilon}$. More serious is the question of strain-thickening versus strain-thinning behavior. If $0 < -\Psi_2/\Psi_1 < 0.5$, then η_3 would be strain thickening, but η_6 would be strain thinning; if $0.5 < -\Psi_2/\Psi_1 < 1$, the opposite behavior is predicted. If $-\Psi_2/\Psi_1 = 0.5$, then η_3 and η_6 would each be strain thinning; η_4 is always predicted to be strain thinning.

Goldsmith Empirical Model. Goldsmith[27] determined high-speed, high-temperature stress–strain data on two proprietary materials, one of which was an amorphous polymer; the other was semicrystalline. Using a high-speed video camera in conjunction with an Instron testing machine, molded ASTM D638 Type M-I tensile bars were pulled at three cross-head speeds and seven temperatures. The data were correlated as

$$\sigma = \frac{C_1[\dot{\varepsilon} - (1/\dot{\varepsilon})^2]}{C_2 + \dot{\varepsilon}} \tag{2.47a}$$

where σ is the engineering tensile stress (based on original cross-sectional area) and $\dot{\varepsilon}$ is the extension ratio, $(L - L_0)/L_0$. He found that for the amorphous material

$$C_1 = (2.07 \times 10^{14}) \exp(-0.048T) \qquad C_2 = 8.33 \ln\left(\frac{413}{T}\right) \tag{2.47b}$$

whereas, for the crystalline material,

$$C_1 = (4.5 \times 10^{11}) \exp(-0.036T) \qquad C_2 = 14.3 \ln\left(\frac{440}{T}\right) \tag{2.47c}$$

where temperature T is in kelvins. This is an empirical analog to the uniaxial extensional viscosity, η_3; compare Eq. 2.8.

The author has seen the high-speed films of Denson[3] in which sheets of polymers exhibited planar extension and uniform biaxial extension. Correlation of his experimental data would yield equations for η_4 and η_6, respectively.

Equations of Motion

The equation of motion is

$$\operatorname{div}\{\eta[\operatorname{grad}(\mathbf{v}) + (\operatorname{grad}(\mathbf{v}))^T]\} - \rho\mathbf{v}\cdot\operatorname{grad}(\mathbf{v}) + \rho\mathbf{g} - \operatorname{grad}(P) = \rho\left(\frac{\partial \mathbf{v}}{\partial t}\right) \tag{2.48}$$

It may have one-to-three nonzero velocity components—the dependent variables; hence, there may be one-to-three differential equations to be solved. These may be functions of one-to-three spatial coordinates and, perhaps, time—the independent variables. If there is only one independent variable, then the differential equation is "ordinary" (ODE); if there are more than one, then the differential equation is "partial" (PDE).

Velocity Profile and Volumetric Throughput

The equations of motion are first solved for the velocity profiles. These are then integrated to obtain the volumetric flowrate. If the velocity is dependent on one direction only, then

$$Q = \int\!\!\int v(x_1)\, dA(x_2,x_3) \tag{2.49}$$

Product Dimensions

For continuous processes, the product dimensions constitute the cross-sectional area of the profile. For cyclic processes, the shape and/or size of the mold halves, and the behavior of the polymer melt, determine the dimensions of the part produced.

Operating Parameters

The operating parameters are those variables that may be changed in order to affect the measure of performance. These include the screw speed (rpm), wire speed (m/s), or roll speed (rpm) for extrusion, wire coating, and calendering, respectively, and injection pressure (Pa) in injection molding.

2.3 Effects of Temperature

Since plastic parts processing inherently consists of shaping pellets or a preform of polymer into a different desired shape, either a polymer melt or softened sheet at a high temperature is converted into a solid object at ambient temperature; hence, these processes are nonisothermal. As discussed below, polymer melts exhibit viscous heating; thus, every plastics parts process is inherently affected by temperature.

Viscous Heating

Inherent in shear flow is the phenomenon of *viscous heating*—the irreversible generation of thermal energy from the transfer of momentum. The magnitude of this generation, and the subsequent rate of temperature rise, are given by

$$\boldsymbol{\tau} : \mathrm{grad}(\mathbf{v}) = \eta\Phi = \rho C_P \frac{dT}{dt} \tag{2.50}$$

For a polymer melt with density near 1000 kg/m^3, heat capacity near 1–2 kJ/kg·°C, and viscosity on the order of 100 Pa·s, subjected to shear rates in the range of 100–1000 s^{-1}, the temperature will rise from 0.5 to 100°C/s.

Thermal Thinning

The effect of temperature on the shear viscosity is described by the Arrhenius equation,

$$\eta(\dot{\gamma},T) = \eta(\dot{\gamma},T_0) \exp\left\{\frac{E_a}{R}\left(\frac{1}{T} - \frac{1}{T_0}\right)\right\} \tag{2.51}$$

where E_a is the activation energy of shear [J/kmol] at $\dot{\gamma}$ and R is the energy constant 8314.4 J/kmol·K.

The Energy Equation

The energy equation is

$$\mathrm{div}[k\ \mathrm{grad}(T)] - \rho C_P \mathbf{v}\cdot\mathrm{grad}(T) + \boldsymbol{\tau} : \mathrm{grad}(\mathbf{v}) = \rho C_P \left(\frac{\partial T}{\partial t}\right) \tag{2.52}$$

The first term is the molecular transfer of thermal energy (heat conduction); its magnitude is determined by the temperature gradient and the thermal conductivity of the polymeric material. The second term is the convective transfer of thermal energy (heat convection); its magnitude depends upon the velocity of the convecting medium and the gradient of temperature in that direction. The third is the viscous heating term, Eq. 2.50. The right-hand side is the unsteady-state term. After solving this equation for the temperature profile, its derivatives yield the magnitudes of thermal energy fluxes (see Chapter 10 of Bird, Stewart, and Lightfoot[1]).

2.4 Effects of Pressure

Usually, it is an acceptable approximation to regard liquids as being "incompressible"; that is, their density changes little with pressure. However, in injection molding of parts whose shape and dimensions are critical, such as compact disks, the effect of pressure, from injection at 10,000–20,000 psi to ejection at atmospheric pressure, must be taken into account.

P–T–V Relationships for Polymer Melts

Although significant advancements have been made in the modeling of the *P–T–V* relationship—an equation of state (EOS)—for polymer melts, the simple one due to Spencer and Gilmore,[28]

$$(P + \Pi)(V - \Omega) = RT \tag{2.53}$$

may be sufficient for engineering purposes. Here, Π and Ω are parameters that permit one to fit experimental data; Π is sensitive to the pressure at which the $V(T)$ are measured, whereas Ω is not—this is a manifestation of the complex relationship between P, T, and V.

Although Tadmor and Gogos[29] list several other models, Capt and Kamal[30] have compared the applicability of the Tait and inverse-volume equations for 12 polyethylene resins, using isobaric and isothermal data. The Tait[31] equation, "originally proposed to explain the compressibility behavior of seawater," is

$$V(P,T) = V(0,T)\left[1 - C\ln\left(1 + \frac{P}{B(T)}\right)\right] \tag{2.54a}$$

$$V(0,T) = \begin{cases} A_0 + A_1T + A_2T^2 + \cdots & T < T_{\mathrm{m}} \quad (2.54\mathrm{b}) \\ V_0\exp(\alpha T) \quad T > T_m & (2.54\mathrm{c}) \end{cases}$$

$$B(T) = B_0\exp(-B_1T) \tag{2.54d}$$

where A_i, B_i, and C are data-fitting constants and α is a constant thermal expansion coefficient. The inverse-volume equation, due to Kamal and Levan,[32] is

$$\rho = \rho_\infty + \left(\frac{\partial\rho}{\partial T}\right)_{P=0} T + (a + bT)P + \left(\frac{c + dT}{2}\right)P^2 \tag{2.55}$$

where ρ_∞ is the density at zero pressure and 0 K and a, b, c, and d are data-fitting constants; $(\partial\rho/\partial T)_{P=0}$ is also an "adjustable parameter."

Pressure as an Operating Variable

Pressure is sometimes an independently adjustable variable that may be used to affect the measure of performance; other times it is not. In extrusion, the pressure developed within the barrel is a dependent variable; it is this pressure that governs the flow of the melt through the die. In calendering, the pressure is also a dependent variable.

However, in wire coating, where drag flow is the primary source of momentum, an imposed pressure on the melt is a valuable independent variable that is used to control the thickness of the coating on the wire. Likewise, in film blowing, the internal pressure is an independent variable that influences the diameter and thickness of the resulting "bubble." Clearly, in injection molding, blow molding, and thermal forming, the pressure influences the rate at which parts are made; this may or may not have a significant influence on the cycle time.

2.5 Extrusion of a Rod as an Example

The extrusion of a cylindrical rod is a continuous process that will serve as an example to synthesize the fundamental concepts discussed above. (The appropriate nomenclature appears in Section 3.)

Accept, without derivation or proof, the equation for the approximate volumetric throughput, Q, from the isothermal extrusion of a Newtonian melt:

$$Q_{\text{extr}} = A'N - \frac{B'P}{\mu} \tag{2.56}$$

where N is the rotational speed of the screw, P is the developed pressure, μ is the viscosity of the melt, and A' and B' are constants. Accept, without derivation or proof, the isothermal Newtonian solution for the volumetric flowrate through a circular hole die as the result of pressure flow, expressed in the form

$$Q_{\text{die}} = \frac{CP}{\mu} \tag{2.57}$$

where C, a constant, is $(\pi D^4)/(128L)$, in which D is the diameter of the hole and L is the length of that hole (the "land" of the die). When equated, since $Q_{\text{extr}} = Q_{\text{die}}$ for an incompressible fluid, Eqs. 2.55 and 2.56 become

$$Q = \frac{A'C}{B' + C} N \tag{2.58}$$

The density of the extrudate is that of the melt, ρ_m; hence, the mass flowrate is $\rho_m Q$.

It is known[33] that the diameter of such an extruded cylinder, as the melt, will be 13% larger than the die opening from which it is extruded. After air cooling (or passing through a water bath), the density of the rod is that of the solid, ρ_s. The rod is produced at the mass flowrate of $\pi D'^2 \rho_s v_s / 4$, where D' is the diameter of the product and v_s is the linear take-up velocity. Hence, for a rod, this velocity is given by

$$v_s = \frac{4}{(1.13)^2} \frac{A'D^2}{128LB' + \pi D^4} \frac{\rho_m}{\rho_s} N \tag{2.59}$$

The take-up velocity necessary to produce a rod of diameter D' is a function of the geometric factors (A', B', D, and L), the material properties (ρ_m and ρ_s, but not μ), and an operating parameter (N). For this and all other *zero-level* solutions (isothermal flow of an incompressible Newtonian melt), there is no interaction between these three categories. That is, changing the screw speed has its influence, which depends neither upon the geometric factors nor the material properties; likewise, changing the temperature, which influences ρ_m and maybe ρ_s, manifests its effect independent of geometric factors or N. This situation is *not* true for either nonisothermal flows or non-Newtonian fluids. In the real world, even the influence of the geometric factors depends upon the material properties and the operating parameters.

One further comment: Accept, without derivation or proof, the equation for the power, ¶, expended in the isothermal extrusion of a Newtonian melt:

$$¶ = Ez\mu N^2 + A'PN = \left(Ez + \frac{A'^2}{B' + C}\right) \mu N^2 = F'\mu N^2 \qquad (2.60)$$

where z is the helical length of the extruder screw and E is a constant; hence, F' is a constant. Note that a 20% increase in the quantity of rod produced is at the expense of a 41% increase, $(1.2)^2 - (1)^2$, in the power required. Hence, the rate of return for sales decreases with increased production.

PART I. Continuous Processes: Shear Dominated

In each of the following processes, the isothermal Newtonian solution for the measure of performance is a function of the relevant geometric factors, material properties, and operating parameters. The more realistic problems are addressed where solutions are available. (The nomenclature for each process appears at the beginning of its section.)

3 EXTRUSION: SINGLE SCREW

Constants and Definitions

$A = \frac{1}{2}\pi mbdw \cos(\theta)\ [1 - (c/b)^2]F_d \approx A'$
$A' = \frac{1}{2}\pi^2 bd^2 \cos(\theta) \sin(\theta)$ if $m = 1$, $c \approx 0$, and $F_d \approx 1$
b = Channel depth of the screw in the metering zone
$B = (\frac{1}{12})(mb^3w/z) \sin(\theta)\ \{1 + (c/b)^3(w/e)[\cos(\theta)\sin(\theta)]^{-2}\}F_p \approx B'$
$B' = (\frac{1}{12}\pi)(b^3d/z) \sin(\theta)$ if $m = 1$, $c \approx 0$, and $F_p \approx 1$
c = Clearance between the screw flights and the barrel
$C = WH^3/12L$, rectangular slit of width W and thickness H
$= \pi D^4/128L$, cylinder of diameter D
$= (\pi D^4/128L)\ f(\kappa)$, concentric cylindrical annulus with outer diameter D and inner diameter $\equiv \kappa D$ where $f(\kappa) = [(1 - \kappa^4) - (1 - \kappa^2)^2/\ln(1/\kappa)]$
$\approx (\pi D^4/192L)(1 + \kappa - \kappa^3 - \kappa^4)$, pipe, if $\kappa > 0.6$
see Tadmor and Gogos[29] for other geometries
d = Inside diameter of the barrel
D' = Diameter of a solidified cylindrical rod; $D' \approx 1.13D$
e = Width of the screw flights
$E = \pi^3(d^3/b) \sin(\theta)[1 + \sin^3(\theta)]$
$F = Ez + A^2/(B + C)$
$F' = Ez + A'^2/(B' + C)$
$F_d = (16w/\pi^3b)\Sigma_{i=1,3,5,\ldots}^{\infty}\ (1/i^3)\tanh(i\pi b/2w)$
$F_p = 1 - (192b/\pi^5w)\Sigma_{i=1,3,5,\ldots}^{\infty}\ (1/i^5)\tanh(i\pi w/2b)$
H' =Thickness of a solidified sheet; $H' \approx 1.20H$
$l = z \sin(\theta)$, axial length of the metering zone; $l/d \approx 10$
L = Length of the die (in the direction of product flow); the "land" of the die
m = number of parallel channels in the screw
N = Screw speed, as "revolutions" per unit time; the "traversed circumference per revolution," πd, has already been incorporated into the screw constants
P = Pressure developed by the screw, the pressure drop across the die
Q = Volumetric flowrate developed by the screw, volumetric flowrate through the die, each of which is for the melt

S = Cross-sectional area of the solid extrudate
v_s = Take-up velocity of the extruded product
V_E = Cost of electricity in customary units, \$/kWh
V_p = Lineal value of the extruded product, \$/ft
V_S = Mass value of the extruded product, \$/lb
w = $(\pi d/m)\sin(\theta)\{1 - (2c/d) - [me/(\pi d \sin(\theta))]\}$, width of the screw channel
z = Helical length of the metering zone channel
θ = Screw helix angle; $\theta = 17.65°$ is a "square pitched" screw, where "lead = diameter"
μ = Newtonian viscosity of the melt
ρ = Density; ρ_m, . . . of the melt; ρ_s, . . . of the solid
¶ = Power

Extrusion is the most important continuous polymer processing operation. An extruder with a die makes a never-ending product with a specific cross-sectional shape. Extruders are also the upstream equipment for the continuous processes of wire coating, calendering, fiber spinning, and film blowing. They are also used to produce the upstream preparts for the cyclic processes of blow molding and thermoforming.

The extruder proper is a rotating screw within a barrel. The diameter of screw thread(s)—there may be more than one set of flights—nearly equals the inner diameter of the barrel; i.e., the clearance is very small. It is the normal stress functions—Eqs. 2.5 and 2.6—of the polymer melt that keep this cantilevered screw centered in the barrel to prevent metal-on-metal friction.

The screw has three zones: (1) Near the feed port, the diameter of the root of the screw is somewhat smaller than that of the barrel, producing a deep channel to accept solid polymer pellets; solid compaction occurs in this zone in which air is removed. (2) Following is a zone in which the diameter of the root of the screw increases considerably; all melting of the pellets must take place within this zone. (3) Finally, there is a metering zone with a shallow channel; within this zone the melt is delivered to the die at a constant rate. Within the metering zone, the pressure increases toward the die.

The turning screw drags—not "pushes"—the melt toward the die; the screw supplies momentum to the melt. But, because the screw flight has a small clearance with the barrel, there is pressure flow of the melt toward the feed throat. The net flow—drag > pressure—supplies melt upstream of the die at a relatively high pressure. Pressure flow then causes the melt to pass through the die, producing an object somewhat like the shape of the die opening.

Shirrell[34] derived the equations for both the isothermal and adiabatic extrusion of a Newtonian melt through an arbitrary die. His results corrected several errors in the version published by Tadmor and Klein.[35] The approximate forms of the isothermal equations have already appeared in Section 2.5; the rigorous ones appear here.

$$Q_{\text{extr}} = AN - \frac{BP}{\mu} \tag{3.1}$$

$$Q_{\text{die}} = \frac{CP}{\mu} \tag{3.2}$$

$$Q = \frac{AC}{B + C} N \tag{3.3}$$

$$\P = Ez\mu N^2 + APN = \left(Ez + \frac{A^2}{B + C} \right) \mu N^2 = F\mu N^2 \tag{3.4}$$

As was done for the rod in Section 2.5, for a given profile one must select a measurable dimension, express its relation to the cross-sectional area, and determine C. Mitsoulis[33] determined that, for Newtonian fluids, the increase in "thickness" of the extruded melt varies from 13% for a solid cylinder ($\kappa = 0$) to 20% for a rectangular sheet ($\kappa \rightarrow 1$; hollow concentric cylinders—pipes—have intermediate values, depending on the nonzero value of κ.

For the non-Newtonian extrusion of a rod, one might use Tanner's equation,[36]

$$\frac{D'}{D} = 0.1 + \left(1 + \frac{(\Psi_1 \dot{\gamma}_{21} / \eta)_w^2}{8} \right)^{1/6} \tag{3.5}$$

where the subscript w means "evaluate this term at the wall conditions." This equation was used by Horwatt[37] in his FORTRAN program to describe the process dynamics and control of the diameter of an extruded rod, with take-up speed as the manipulated variable.

Abdel-Khalik et al.[38] extended the utility of the Tanner equation by developing a relationship between Ψ_1 and η, based on the Goddard-Miller rheological equation of state (see Ref. 2, 1st. ed., Art. 7.5).

The material choice and temperature of extrusion will determine μ, ρ_m, and ρ_s. The desired size of the extrudate determines v_s. Analogous to the approximate solution given in Eq. 2.58, for a rod the take-up velocity is given by

$$v_{s,\text{rod}} = \frac{4}{(1.13)^2} \frac{AD^2}{128LB + \pi D^4} \frac{\rho_m}{\rho_s} N \tag{3.6}$$

Similarly, for a sheet the take-up velocity is given by

$$v_{s,\text{sheet}} = \frac{1}{1.20} \frac{AH^2}{12LB + WH^3} \frac{\rho_m}{\rho_s} N \tag{3.7}$$

In general,

$$v_{s,\text{extrudate}} = f(\text{screw } A \text{ and } B; \text{ die dimensions}) \frac{\rho_m}{\rho_s} N \tag{3.8}$$

The selection of an extruder—barrel and screw—to accomplish this task will determine A, B, and E. The capability of that combination will determine P.

Since an extruder is not a positive-displacement pump, it can be operated with a blank die (one that has no opening); i.e., $Q_{\text{extr}} = 0$. The resulting pressure is the maximum that may be developed:

$$P_{\max} = \frac{A}{B} \mu N \tag{3.9a}$$

Operating the extruder without a die ($P = 0$) yields the maximum throughput:

$$Q_{\max} = AN \tag{3.9b}$$

Obviously, $Q < Q_{\max}$ and $P < P_{\max}$. The conditions at which one should operate a given extruder-die combination requires the customary optimization of the value of the extrudate and its production cost. Considering only the "sales" value of the extrudate versus the cost of the electricity to turn the screw,

$$N_{\text{opt}} = \frac{AC\rho_m V_S}{2[A^2 + (B + C)Ez]\mu V_E} \tag{3.10a}$$

or

$$N_{\text{opt}} = \frac{AC(\rho_m/\rho_s)(V_P/S)}{2[A^2 + (B + C)Ez]\mu V_E} \tag{3.10b}$$

In these equations, consistent units, or conversion factors, must be used.

Shirrell's[34] solution of the adiabatic case was accomplished using a Runge–Kutta numerical iteration. The three equations for flowrate, power requirement, and temperature rise were

$$\frac{dP}{dz} = (A'N - Q)\frac{\mu}{B'} \tag{3.11}$$

$$\frac{d\P}{dz} = E\mu N^2 + A'N\frac{dP}{dz} \tag{3.12}$$

$$\frac{dT}{dz} = \left(\frac{1}{CQ}\right)\left(\frac{d\P}{dz}\right) + \left(\frac{1}{C}\right)\left(\frac{dP}{dz}\right) \tag{3.13}$$

with

$$\mu = \mu_0 \exp[-\beta(T - T_0)] \tag{3.14}$$

He[34] also modeled the screw extruder as a polymerization reactor for styrene; his program was written in Basic. Ray[39] developed an improved FORTRAN version for the free radical, thermally initiated, polymerization of azoisobutyronitrile.

Chiu[40] has written FORTRAN programs for the pressure flow of Newtonian and Carreau fluids through three different die shapes—a cylinder, a cylindrical annulus, and a rectangular conduit. Three different thermal conditions were considered: isothermal, constant wall temperature, and adiabatic. The density, thermal conductivity, overall heat transfer coefficient, low-shear-rate-limiting viscosity, as well as the Carreau parameter, may be temperature dependent. These programs solve for the velocity and (for the nonisothermal cases) temperature profiles.

Ishmael[41] has modeled the postextrusion cooling and freezing process as the extruded melt is turned into a solid rod and chopped into pellets. The successive heat transfers are from the extrudate to (i) air, (ii) water, (iii) air, (iv) puller-rollers, and (v) air; this sequence is followed by the pelletizer. Required data include the polymer density, viscosity, thermal conductivity, heat capacity, and thermal expansion, each as a function of temperature.

The model predicts the length of the water bath, the final temperature, and the final inner and outer diameters of the extruded strands. These strands are hollow because the outer surface is frozen first, and subsequent cooling and contraction creates a vacuum on the inside. The model is applicable to crystalline and amorphous polymers; it was confirmed by industrial experiments with isotactic and atactic polypropylene. Although Ishmael[41] used spreadsheet software, the equations are given and permit one to use an alternative means of calculation.

Cecil[42] showed that repeated processing, as experienced by "regrind"—otherwise scrap material chopped into pellets and sent back to be reextruded—in the feed, affected the color stability of extruded parts. (Reprocessing also occurs in injection molding, *vide infra*.) Cecil measured the yellowness index (YI) of five grades of polypropylene, without and with various additives, subjected to two or five additional passes through an extruder. The results showed that: (1) repeated processing increased the YI; (2) small amounts of antioxidant—0.10% for two additional "cycles" and 0.05% for five—reduced the YI, but larger amounts increased it; (4) other changes in the recipe, such as the inclusion of a filler deactivator, a secondary stabilizer, and/or pigment, also had an affect on YI.

4 EXTRUSION: TWIN SCREW

Twin-screw extruders are constructed in a variety of ways: they may be corotating (in the *same* clockwise or counterclockwise direction) or counterrotating (*opposite* directions); they may be nonintermeshing (which means that looking in the axial direction, the flights of the two screws never overlap), partially intermeshing, or fully intermeshing. (The book by Janssen, cited in the bibliography, shows examples of these configurations on pp. 6–9; the book by Martelli, also cited in the bibliography, has a more complete classification scheme on pp. 9–12.)

Tolliver[43] successfully modeled a vented nonintermeshing counterrotating twin-screw extruder used for the final polymerization of an industrial polymer. The screws were 6 in. in diameter and 50 ft. long, followed by a short single screw that produced the extrudate. His FORTRAN program requires knowledge of (a) the type of element, its flight land width, its volume, and its cross-sectional area, (b) the screw geometry data (barrel diameter, flight clearance, and helical angle), and (c) locations of the heating zones. "Rework and additive ports are defined in separate [program] statements."

Operating conditions to be specified include initial flow rate, melt temperature, reactor yield, pressures, screw speed, vent/additive/rework flowrates, and zone temperatures. Physical property data required are density, viscosity, heat capacity, heat of vaporization, and monomer vapor pressure and temperature. The program calculates the volumetric flowrate, temperature distributions, and power consumption. Prior work on this modeling effort was done by Hudson[44]; Richardson[45] had previously modeled a similar partially intermeshing twin-screw extruder.

5 WIRE COATING

Constants and Definitions

$A = \frac{1}{2}PR/\tau_{1/2}$
$a_1 = \frac{1}{2}c(\beta^2 - 1 - \ln \beta^2)$
$a_2 = \frac{1}{2}c(\beta^2 - 1 - \beta^2 \ln \beta^2)$
$B = R\eta_0\tau_{1/2}$
$b = \frac{1}{2}c \ln \beta^2 + c^\alpha(\beta^{\alpha-1} - 1)/(\alpha - 1)$
$C = [A(1 - k^2) - v/B]/\ln k^2$
$c = [b - c^\alpha(\beta^{\alpha-1} - 1)/(\alpha - 1)]/\ln \beta$, which is implicit in c
$c_{\mathrm{ED}} = \{a_1 c + [2c^\alpha/(\alpha - 1)][(\beta^2 - \beta^{\alpha-1})/(3 - \alpha) - \frac{1}{2}(\beta^2 - 1)]\}(\rho_m/\rho_s)$
$c_{\mathrm{E3D}} = [(a_1 c - c^3 a_2)/b](\rho_m/\rho_s)$
$c_{\mathrm{ND}} = [(\beta^2 - 1)/\ln(\beta^2)](\rho_m/\rho_s)$
$c_{\mathrm{PLD}} = \{(\beta^2 - 1)/(1 - \beta^{(1-n)/n}) + [2n/(3n - 1)](1 - \beta^{(3n-1)/n})\}(\rho_m/\rho_s)$
D = Diameter of the die
d = Diameter of the wire
$k = (d/D) < 1$
P = Pressure in the melt, upstream of the die
R = Radius of the die $= \frac{1}{2}D$
v = Velocity of the wire
$\beta = (1/k) = (D/d) > 1$
δ = Thickness of the coating
η_0 = Zero-shear-rate-limit viscosity of the melt
$\lambda = \sqrt{(1 - k^2)/\ln(\beta^2)}$
ρ_m = Density of the polymer melt
ρ_s = Density of the solidified coating
$\tau_{1/2}$ = Ellis model parameter

In the usual wire coating operation, a metal wire is drawn through a polymer melt and the combined product passes through a die; the polymer is subsequently solidified around the wire to provide a suitable electrical insulation. An extruder upstream provides a sufficient quantity of polymer melt.

As mentioned earlier, this process is an example of drag flow; the momentum of the moving wire is transferred to the polymer, causing it flow through the die. The first normal stress function of the melt keeps the wire centered within the surrounding polymer. Convective heat transfer from the ambient air removes the latent heat of fusion of the polymer, and the solidified product is wound on spindles for distribution.

5.1 Drag Flow Only

For drag flow only, the melt pool must come from a reservoir that is uninfluenced by the upstream extruder. When one solves this problem for drag flow of a Newtonian melt, a very limiting solution is obtained: the thickness of the coating is a function of the die and wire dimensions *only*!

$$\frac{\delta}{d} = \frac{\sqrt{1 + c_{\text{ND}}}}{2} - 1 \tag{5.1}$$

In one sense, this solution is appealing: The thickness is predicted to be independent of wire speed, polymer melt properties, temperature, etc. However, knowing that process upsets will occur, this result gives no indication of how to control the coating thickness. And, only one product can be made for a given die and wire; there is no process flexibility.

For a power law fluid (see Section 2.2),

$$\frac{\delta}{d} = \frac{\sqrt{1 + c_{\text{PLD}}}}{2} - 1 \tag{5.2}$$

Boese[46] has solved these problems for an Ellis fluid (see Section 2.2), using the assumption that $\rho_s \approx \rho_m$; the equations given here do not make that assumption. Three cases exist: $\alpha = 1$ is the Newtonian solution, Eq. (5.1); $\alpha \neq 1$ or 3 is the general Ellis case; finally, $\alpha = 3$ is a special Ellis case. For $\alpha \neq 1$ or 3,

$$\frac{\delta}{d} = \frac{\sqrt{1 + c_{\text{ED}}} - 1}{2} \tag{5.3}$$

For $\alpha = 3$, (5.4)

$$\frac{\delta}{d} = \frac{\sqrt{1 + c_{\text{E3D}}} - 1}{2}$$

5.2 Drag and Pressure Flow

To overcome the limitations of drag flow only, pressure flow is added by causing the pressure in the melt pool to be that developed by the extruder. Now, the coating thickness is a function of variables that provide a means of process control and a variety of products from a given wire and die.

There are two possible situations. If the maximum velocity of the melt around the wire is that of the wire, then drag flow is controlling, even though pressure flow is present. If, however, the maximum velocity in the melt occurs between the wire and the die, then pressure flow is controlling, even though drag flow is present.

For drag flow controlling,

$$\frac{\delta}{R} = \sqrt{\frac{B}{v}\left[\left(\frac{A}{2}\right)(1 - 2k^2 + k^4) + C(1 + k^2 \ln k^2 - k^2)\right] + k^2} - k \qquad (5.5a)$$

For pressure flow controlling,

$$\frac{\delta}{R} = \sqrt{\frac{AB}{v}\left(\frac{1 - 4\lambda^2 + 4\lambda^2 k^2 - k^4}{2} + \lambda^4 \ln k^2\right) + \lambda^2} - k \qquad (5.5b)$$

These equations are expressed in terms of the Boese[46] parameters to facilitate the use of his graphical results.

6 CALENDERING

Constants and Definitions

F = Force trying to separate the second and third rolls of a three-roll stack
H = Thickness of the calendered sheet
H_f = Thickness of the melt fed between the second and third rolls
H_0 = Closest approach distance between the second and third rolls, the nip
P = Pressure within the melt
Q = Volumetric flowrate of the calendered sheet
R = Radius of the rolls
T = Temperature within the melt
V = Tangential velocity of the second and third roll surfaces
W = Width of the calendered sheet
x = Distance from the nip, in the direction in which the sheetis being calendered
X = Dimensionless "distance" measured from the nip, $x/\sqrt{(Rh_0)}$
λ = Dimensionless "distance" where the calendered sheet leaves the rolls

In calendering, an extruder supplies a sheet of polymer melt of width W that is placed on top of a heated clockwise-rotating cylinder, a roll. Below this is a second roll, rotating counterclockwise; if it rotates slightly faster than the first, the melt will leave the first and follow the second. Finally, there is a clockwise-rotating third roll. The sheet passes between the latter two, whose line of closest approach is called the nip.

Within the region where the sheet contacts both the second and third rolls, the pressure within the melt rises and falls; this must be the case, since the upstream and downstream pressures are ultimately ambient. Although calendering contains drag and pressure flow, the former dominates; the melt receives its momentum primarily from the metal rolls.

To produce a uniform thickness across the width of the sheet, the axes of the rolls need to be skewed; the gap between the rolls needs to be smaller in the middle of the sheet and larger at the sides. This, again, is a requirement due to the normal stress functions of the polymer.

Ewing[47] derived the equations that relate the thickness of a calendered sheet to the processing variables for the isothermal processing of a Newtonian melt, a power law fluid, a Bingham plastic, and special case ($n = 0.5$) of the Cross model (see the generalized Yasuda model in Section 2.2). For a Newtonian melt,

$$H = (1 + \lambda^2)H_0 \tag{6.1}$$

If the feed to the last pair of rolls is unlimited in thickness, then $\lambda = 0.47513$; hence,

$$H = 1.2257H_0 \tag{6.2}$$

If, however, the feed to the last pair of rolls is finite, and not much different than the nip, then λ is a function of that thickness. The results show that λ decreases significantly for $H_f/H_0 < 10$.

Although simple, these results are disturbing because they predict that the thickness of the calendered sheet is independent of everything but the gap between the last two "finishing" rolls. If true, then the only way to control the thickness of the product to be sold is to control the position of these large fast-rotating rolls of metal.

Since "real" melts are non-Newtonian and possess normal stress functions (see Section 2.2), these forces try to separate the rolls; hence, control of the thickness of the calendered sheet is best accomplished by adjusting the "roll separating force" rather than the size of the nip.

For a shear-thinning power law fluid, $0.47513 < \lambda < 0.522$ and $1.2257 < H/H_0 < 1.295$ as $1 > n > 0.2$, respectively.

For the Cross model, the equations were "unsolvable" except for the special case of $n = 0.5$. For the Bingham plastic, a solid plug exists in the feed, between the rolls, and (of course) as the calendered sheet. The last region exists not because the sheet has solidified but because the shear stress is less than τ_0. An example shows that disjoint regions of the shear-thinned melt may exist between the feed and the nip; these are separated by a solid plug.

PART II. Continuous Processes: Extension Dominated

7 FIBER SPINNING: MELT

Fiber spinning processes include melt, dry, and wet. In melt spinning, a polymer melt is extruded through a spinneret and the strands are cooled and solidified in air; heat transfer is the only transport phenomenon. In dry spinning, a polymer solution is extruded and the solvent is evaporated to produce solid strands; here, both heat and mass transfer are involved. In wet spinning, a solution is extruded and the solidification involves mass transfer of the solvent from the strands in a coagulation process; hence, mass transfer to and from the fiber occurs.

McMackin[48] developed and compared empirical models for melt spinning, using experimental data from an industrial laboratory. She developed 12 models for poly(ethylene-terephthalate), which describe how stick point, tension, and denier per filament (dpf) vary with melt temperature, winder speed, and pump speed. The parametric variables were spinneret hole size and intrinsic viscosity (which was used to characterize the polymer, even though this was *melt*, not dry or wet, spinning).

McMackin[48] concluded that (1) stick point depended primarily on pump speed, (2) tension depended primarily on winder speed, and (3) dpf was influenced by pump speed, winder speed, their interaction, and melt temperature.

8 FILM BLOWING

Constants and Definitions

A_n = Multiplicative constant of I_1 in the infinite series
B_n = Multiplicative constant of K_1 in the infinite series
c = $(Q_{\text{ann}}/\pi V_F)(\rho_m/\rho_s)$
I_1 = Modified first-order Bessel function of the first kind
K_1 = Modified first-order Bessel function of the second kind
P = Pressure of the air inside the bubble
r = Radial position within the film
R = Outside radius of the film at z
R_D = Outside radius of the die
R_F = Outside radius of the film at the frost line
V_D = Average velocity of the melt at the exit of the die
V_F = Constant velocity of the solid film at the frost line
z = Axial position; $z = 0$ at the die
Z_F = Axial position of the frost line
δ = Thickness of the film
δ_F = Thickness of the film at the frost line
κ = Inside radius of the annular die/R_D
ρ_m = Density of the polymer melt
ρ_s = Density of the solidified film
ψ = Stream function

Film blowing is the process whereby a relatively thick-walled small-radius annulus is converted into a relatively thin-walled large-radius annulus. The polymer melt is extruded from an annular die containing a center hole through which air pressure provides the source of momentum to expand the polymer film. Outside air-cooling causes the molten film to solidify; this occurs at the frost line. The solid film is collapsed and drawn through nip rollers, which maintain the pressure (with little-to-no air leakage).

A material balance between the die and the frost line leads to the relationship

$$\delta_F = \left[(1 - \kappa^2) \frac{1}{2} \frac{R_D^2}{R_F} \right] \frac{V_D}{V_F} \tag{8.1}$$

Hence, to make a bag with a given circumference (or a flat sheet with a given width), $2\pi R_F$, and a specified thickness, δ_F, from a given die (with dimensions R_D and κR_D), the extruder output must be

$$Q_{\text{die}} = (1 - \kappa^2) R_D^2 V_D \tag{8.2}$$

the same relationship as for the extrusion of a pipe.

Alternatively,

$$\frac{\delta}{R_F} = 1 - \sqrt{1 - c} \tag{8.3}$$

similar to the relations seen for drag-flow wire coating.

Although these results are useful, from them we learn nothing about the shape of the film nor how its size is related to the rheological properties of the melt and the pressure inside the bubble.

It is easy to see that a single cosine function,

$$R = \frac{1}{2}\left[(R_F + R_D) - (R_F - R_D)\cos\left(\frac{\pi z}{Z_F}\right)\right] \tag{8.4}$$

satisfies the boundary conditions at the die and the frost line. However, it is not a solution to the equations of motion. This is a two-dimensional flow problem, which may be expressed in either cylindrical or spherical coordinates:

$$\mathbf{v} = (v_r, 0, v_z) = C(r, 0, z) \tag{8.5a}$$

or

$$\mathbf{v} = (v_r, v_\theta, 0) = S(r, \theta, 0) \tag{8.5b}$$

Sukadia[49] modeled the film blowing process using the stream function in cylindrical coordinates approach (see Section 4.2 of Bird, Stewart, and Lightfoot[1]). In steady-state flow, a constant value of the stream function traces out the path that an element of the fluid follows—the "particle path"; hence, $\psi(r, z)$ describes the shape of the film.

$$\psi(r,z) = \sum_{n=1}^{\infty} r\left[A_n I_1\left(\frac{n\pi r}{Z_F}\right) + B_n K_1\left(\frac{n\pi r}{Z_F}\right)\right]\cos\left(\frac{n\pi z}{Z_F}\right) \tag{8.6}$$

where the A_n and B_n are determined from the orthogonality relationships, evaluated at the die:

$$A_n = \frac{\displaystyle\int_{\kappa \mathrm{RD}}^{\mathrm{RD}} \psi(r,0) I_1(n\pi r/Z_F)\, dr}{\displaystyle\int_{\kappa \mathrm{RD}}^{\mathrm{RD}} r[I_1(n\pi r/Z_F)]^2\, dr} \tag{8.7}$$

and

$$B_n = \frac{\displaystyle\int_{\kappa \mathrm{RD}}^{\mathrm{RD}} \psi(r,0) K_1(n\pi r/Z_F)\, dr}{\displaystyle\int_{\kappa \mathrm{RD}}^{\mathrm{RD}} r[K_1(n\pi r/Z_F)]^2\, dr} \tag{8.8}$$

provided

$$\int_{\kappa \mathrm{RD}}^{\mathrm{RD}} r \left[I_1\left(\frac{n\pi r}{Z_F}\right)\right] \left[K_1 \left(\frac{n\pi r}{Z_F}\right)\right] dr = 0 \tag{8.9}$$

for each n. One can see that

$$\psi(R_D,0) = \psi(R_F,Z_F) \qquad \psi(\kappa R_D,0) = \psi(R_F - \delta,Z_F) \tag{8.10}$$

PART III. Cyclic Processes: Shear Dominated

9 MELT INJECTION MOLDING

Constants and Definitions: Center-Gated Part with Rotational Symmetry

B = Bottom and wall half-thickness
L = Wall length
P = Injection pressure
R = Bottom radius
R_g = Gate radius
t_{fill} = Cavity fill time
μ = Newtonian melt viscosity

Melt injection molding is the cyclic analog to extrusion. It uses a similarly designed screw in a barrel but uses a nozzle instead of a die; this is called the "press." The mold, called the "tool," consists of a sprue bushing, runners, gates, and cavities through which, and into which, the polymer melt is injected in order to produce the desired part; it has (at least) two halves that, when separated, allow one to remove the part.

To produce the polymer melt for the molded parts cyclically, instead of continuously, the screw reciprocates. That is, the screw rotates and moves backward from the nozzle, producing a polymer melt pool in front the screw called the "shot." This pool has more than enough melt to make the next part; the excess must be sufficient to fill the runners, the sprue, as well as providing a cushion of polymer to prevent the metal screw from banging into the end of the barrel upon injection.

If the mold is closed and empty, the screw "rams" forward, injecting the polymer into the tool; it continues to apply pressure, and a little more material, as the melt in the cool mold solidifies. As soon as the gate of the cavity freezes, no melt may come out, and the screw may begin its plasticating step of preparing the next shot. When the part in the cavity is sufficiently cool, such that upon removal it retains its shape, the mold halves open and the part is ejected. Then, the mold halves close, and the cycle is repeated.

Richards[50] used a mold that produced a 2 × 2 in. plaque for Gardner impact (ASTM D3029) and two Type L tension-impact specimens (ASTM D1822) of Lexan 141. This was mounted on a highly instrumented 18g Battenfeld injection molding machine. The injection controls permitted constant velocity injection, rather than the usual constant pressure injection. The viscosity of the Lexan 141 was fully characterized via capillary rheometry, using an Arrhenius dependence (see Section 2.3). The density of each plaque was determined via water displacement.

A complete analytical solution to the plaque filling step was obtained by dividing the problem into (i) diverging flow in a cylindrical wedge for the fan gate and initial entry into the plaque cavity, followed by (ii) rectangular flow between parallel plates for the remainder of the cavity. The resulting velocity profiles were necessary to determine the shear rate profiles, from which the viscosities were specified.

Richard's[50] primary interest was in comparing destructive and nondestructive properties of the parts in order that the latter might be used instead of the former. Plaque volume and weight revealed "global" variations in density, part to part. Optical birefringence revealed local variations within each part due to process orientation. The attempt to use dielectric properties was unsuccessful because the electrodes were the same size as the plaque; hence, no detail "within a part" could be obtained.

In preparation for further experimental studies, Moore[51] and Hudson[52] determined the rheological properties of a single lot of high-density polyethylene (HDPE). O'Nan[10] used these data to compare the ability of various shear viscosity models to describe the shear rate and temperature dependencies. Peterson[53] contributed additional data and correlations using the Arrhenius equation.

Taghizadegan[54] then carried out an extensive experimental study of the ASTM plaque and tensile bar mold used in the Richards dissertation with the HDPE characterized by Moore,[51] Hudson,[52] and Peterson.[53] Utilizing a "composite design" with 16 treatment combinations—eight corners of a cube plus six star points and a replicated center point—response surface analysis of the data yielded equations that related processing conditions to part thickness as well as tensile and impact strength. Then, computer simulations demonstrated the potential effectiveness of employing statistical process control (SPC) as part of a feedback loop to decrease part-to-part variations. This was an extension of prior work by the team of Wright,[55] Ralston et al.,[56] Stoll, and Harper. Lee[57] continued the application of SPC by using the correlations discovered by Taghizadegan.

Henz[58] simulated the cooling phase of the injection-molding process and verified the model experimentally. He used a finite-element program called C-COOL, developed by AC Technology of Ithaca, New York (now dba Mold-Flow, Louisville, Kentucky), to predict the cooling of a rectangular plaque, 21 × 11 × 0.1 in. The experimental phase used polystyrene and 20% talc-filled polypropylene. Henz concluded that the predicted temperature distribution in the part was within 10% of the measured values. The wall temperatures of the mold were less accurately predicted; he attributed this to some imperfect assumptions in the software that "should be . . . easy to remedy."

Barbarito[59] showed that the hold pressure affected the impact strength of thermally aged polycarbonate plaques. Notched Izod specimens were aged in a silicone oil bath at 110°C, and slow cooled them before testing. For a lower hold pressure of 250 psi, the ductile-to-brittle transition ran from 10 to 29 min, whereas, for a higher hold pressure of 1000 psi, this transition was delayed as long as 40 min. The average transition times were 15.3 and 16.2 min for the low and high values, respectively.

In Section 3 the work of Cecil[42] was cited to point out that reprocessing material, as in the pelletizing and reuse of the sprue and runners, can affect the properties of the final parts. Experimental studies with total recycling of molded parts are often used to determine the percentage of acceptable regrind. That is:

(1) Properties of a sample of the first "virgin" parts are measured; (2) all of the remaining parts are pelletized and remolded for, perhaps, five cycles, and the properties of a sample of these parts are measured; (3) step 2 is repeated; etc. Then, these measurements are plotted versus the number of cycles; when the data fall below those which are acceptable, the corresponding number of cycles is the percentage of permissible regrind.

Benkert[60] has derived an equation for the fill time of the isothermal injection molding of a solid-walled center-gated part with rotational symmetry, using a Newtonian melt under constant pressure:

$$t_{\text{fill}} = \frac{3\mu}{P}\left(\frac{R}{B}\right)^2 \left[\frac{(L/R)^2}{2} + \left(\frac{L}{R} + \frac{1}{2}\right) \ln\left(\frac{R}{R_g}\right) - \frac{1 - (R_g/R)^2}{4}\right] \tag{9.1}$$

The cavity for a 6.5-lb conical waste receptacle, made of plasticized poly(vinyl chloride), with a maximum diameter of 15.5 in. and a height of 30 in. requires about 10 s to fill.

The selection of a machine to accomplish this task requires one to specify the following recommended heuristics for the "press":

1. The clamping force (tons) shall exceed two times the projected area of the part (in.^{2}). (*Note:* The relationship between these two magnitudes actually depends on the first normal stress function; see Section 2.2. When sufficient data for Ψ_1 are available for a number of polymers, this heuristic will be improved.)
2. Shot size and plasticating capacity are always expressed in ounces of polystyrene (PS); hence, one must calculate these using the ratio of the densities of PS and that of the material being used.
3. Tie-bar separation must be greater than the size of the part (at least in one direction, horizontal or vertical).
4. Open daylight must be greater than twice the part depth plus the closed mold thickness.

A number of commercial injection-molding machines are listed, with these characteristics, in the annual editions of the *Modern Plastics World Encyclopedia* (cited in the bibliography under **Journals,** *Modern Plastics*). Upon finding several that could do the job, a selection would be made on the basis of capital investment and operating expenses.

Although melt injection molding is used overwhelmingly for thermoplastic polymers, it is possible to injection mold thermosetting resins. The author[61] used the same Battenfeld press and ASTM mold as Richards[50] to make solid plaques and tensile bars out of ethylene-propylene-diene monomer (EPDM) elastomers. He found that successful manufacture of such parts, avoiding short-shots and overpacking, was extremely sensitive to injection velocity! In this case, the molds were heated rather than cooled. (Compare reaction injection molding, which produces foamed parts.)

The solid objects produced by melt injection molding usually have relatively thin walls. If the desired object as designed would have thick walls, one can save material costs and produce thin walls by using the new technology of "gas-assist" injection molding. (Again, compare reaction injection molding.)

10 REACTION INJECTION MOLDING

In reaction injection molding, the polymer melt contains a blowing agent, which may be (i) a dissolved gas that flashes upon a reduction of pressure, (ii) a volatile liquid that boils upon an increase of temperature, (iii) a nonvolatile liquid that thermally decomposes, or (iv) a product from a change in the polymer composition. The first two classes are physical blowing agents (PBAs); the third is chemical blowing agents (CBAs). (An extensive list of these agents may be found in the annual issues of *Modern Plastics World Encyclopedia*.) An example of the fourth class is the release of CO_2 from the product of an isocyanate and an acid, which produces an internally foamed polyamide:

$$\begin{aligned}&\text{—R—N}{=}\text{C}{=}\text{O} + \text{HO—CO—R}'\text{—} \rightarrow\\ &\qquad \text{—[R—NH—CO—O—CO—R}'\text{]—} \rightarrow\\ &\qquad\qquad \text{—[R—NH—CO—R}'\text{]—} + CO_2\end{aligned}$$

In any case, the injection takes place under much lower pressures than those of melt injection molding (IM). Then, the use of a heated mold (rather than a chilled mold, as in IM) causes the volatilization of a physical blowing agent, the decomposition of a chemical blowing agent, or a reaction (such as that shown above) to take place. Obviously, every part that utilizes such an agent or reaction has either closed or open voids. This technology facilitated the creation of foamed plastics that matched the density of wood. Knocking on such objects created the illusion that the object was made of wood.

11 TRANSFER MOLDING

Transfer molding uses thermosetting resins to make an object of intricate geometry, such as a household electrical wall socket. (Compare compression molding, which is used for an object of very simple geometry.) However, to illustrate the complexity of modeling the process, consider a simple plaque mold.

Shah[62] developed six levels of models for the transfer molding of a rectangular parallelepiped with a phenolic resin. Using a recipe from Sauers,[63] data from Mark and Bikales,[64] a software package called FLUENT, as well as the spreadsheet Excel, the various models were (i) isothermal; (ii) nonisothermal, including viscous heating but neglecting $\mu(T)$ and $k(T)$, steady-state heat transfer to constant temperature walls; (iii) as in (ii) but with unsteady-state heat transfer; (iv) . . . including $\mu(T)$; (v) using FLUENT: Constant Pressure; and (vi) FLUENT: Free Surface Model. Velocity and temperature profiles are presented as functions of gap and flow direction distances; in addition, color presentations reveal the variations in temperature. Fill-time predictions are given. What remains for the process engineer is to model the kinetics of cross-linking in the phenolic resin that occurs after fill; the "hold time" before the mold may be opened and the part removed depends upon this analysis, which is a function of the temperature profiles.

PART IV. Cyclic Processes: Extension Dominated

12 COMPRESSION MOLDING

Compression molding uses thermosetting resins to make objects with simple geometry. (Compare transfer molding, which is used for parts with intricate geometries.) Sonta[65] molded a simple rectangular parallelepiped out of sheet molding compound (SMC). He followed the curing process of this material by using dynamic dielectrometry in a specially constructed compression mold; the mold halves had to be electrically insulated from each other. High-speed data acquisition permitted Sonta to follow these reactions, which were over in 40 to 60 s. Hence, as in casting, this technology may be used to increase the rate of production by replacing fixed time schedules such as ''cure for *t* seconds or minutes,'' with curing times based on objectively measured data. (Ramanathan[66] studied this application of dynamic dielectrometry as well as slower reactions observed with epoxies.)

Logsdon[67] modeled the compression molding of a cafeteria tray, also made from SMC. In addition, he did experimental work on the squeezing flow problem, using a sample of Silly Putty (polydimethylsiloxane) in an Instron 4505. If the preform is a cylinder, e.g., $D = H$, then the squeezing operation causes the sides of the cylinder to become curved. The shape of this free surface is determined by the surface tension of the polymer. In addition, this process is a manifestation of the uniform biaxial extensional viscosity of the polymer. Hence, these data are necessary to accurately relate the compression stroke to the mold-filling process.

13 BLOW MOLDING

Constants and Definitions

D_M = Diameter of the mold cavity
D_P = Diameter of the parison
H_M = Height of the mold cavity
R = Radius on the free film
R_M = Radius of the mold cavity
R_P = Radius of the parison
V = Volume of the film
V_P = Volume of the parison
W = Wall thickness of the molded object
$W_{\max}$ = Maximum wall thickness of the molded object
W_P = Wall thickness of the parison
X = Distance down the side wall, measured from the horizontal center plane

Blow molding is used to make a hollow object whose opening is smaller than the outside dimensions. It begins with the cyclic extrusion of a parison, the vertical extrudate from a concentric or eccentric annular die. The mold halves close around the parison, pinching off the bottom. Gas enters the hollow center of the parison and blows it against the insides of the mold. When the part is sufficiently cooled, the mold is opened and the part removed. This is the usual method for making bottles, most with molded threads for caps, some with hollow handles, the latter of which may be contiguous (as in milk and juice bottles—concentric openings) or noncontiguous (as in the microwave reheatable syrup bottles—eccentric openings). The innovation of the programmable parison makes it possible to obtain a controllable nonuniform wall thickness, providing or minimizing squeezability.

Riney[68] modeled the blow molding of a concentric cylindrical bottle from a uniformly thick parison. A volume balance, similar to that employed in the analysis of thermoforming, yields the approximation

$$W_{\max} = W_P \left(\frac{D_P}{D_M} \right) \quad \text{if } D_M = H_M \tag{13.1}$$

or

$$W_{\max} = \frac{W_P R_P}{\sqrt{\{[\frac{1}{2}(\frac{1}{4}H_M^2 - R_M^2)/R_M]^2 + \frac{1}{4}H_M^2\}}} \quad \text{if } D_M < H_M \tag{13.2}$$

This thickness occurs at the vertical center of the molded object; the remaining free film is like a pointed football. (Note that the parison does not move sideways as a cylinder, as implied by Middleman[69] in Problem 3-24.) Riney[68] has written an Excel spreadsheet to calculate the wall thickness of the blow-molded object, from the initial contact point to the last (which occurs in the bottom and top edges). She also attempted to obtain analytical solutions using Maple but was unsuccessful.

14 THERMOFORMING

Constants and Definitions

A = Dimensionless numerator mold constant
B = Dimensional denominator mold constant (length)
C = Dimensionless denominator mold constant
h = Thickness of the free film
h_f = Thickness of the frozen film
h_i = Thickness of the frozen film at the initial touch
h_0 = Thickness of the original preform
R = Radius of the free film, prior to it touching the mold
R_f = Radius of the free film
x = Distance from the center of a mold outward toward the side
z = Slant height, distance down the wall of the mold
Z = Slant height of the mold

Thermoforming is used to make relatively thin-walled objects with simple or complex surfaces, provided undercuts are absent or small. The starting material is generally a flat sheet of uniform thickness; it may be a homogeneous sheet produced by extrusion or a multilayered sheet by "co-extrusion."

Normally, this sheet is warmed from above by radiant heaters; its own weight causes it to sag. A "reverse blow"—air from below—may be used to further stretch the sheet, the once-again-concave sheet is indexed over a female mold, its edges are clamped around the mold, and a vacuum is created between the mold surface and the sheet. This last step draws the sheet against the "cold" mold surface, causing the sheet to solidify.

Among the many variations of thermoforming are drape molding, which is the use of a male mold, and mechanical assist, the use of a wooden plunger covered with mole skin to aid the sheet in reaching deep portions of a mold. The initial heating may be selective by using screens to absorb the radiant heat above areas that are to remain relatively thick or do not need to stretch very much.

An inherent characteristic of thermoformed objects is their nonuniform wall thickness. To the author's knowledge, the only shape not subject to this result is the hemisphere. If a circular sheet of thickness h_0 is clamped above a "hemispherical mold," the heated free film being thermoformed is a portion of a hollow sphere; this film touches the hemispherical mold at a single instant, which, upon freezing, has a uniform thickness. This thickness, h_f, determined from a volume balance, is $\frac{1}{2}h_0$, as shown below:

$$\begin{aligned} V &= \pi R_0^2 h_0 \\ &= \tfrac{1}{2}(\tfrac{4}{3})\pi[R_f^3 - (R_f - h_f)^3] \qquad (14.1a) \\ &= \tfrac{1}{2}(\tfrac{4}{3})\pi[3(R_f - h_f)R_f h_f + h_f^3] \end{aligned}$$

Since $R_f >> h_f$, then $R_f^2 h_f >> h_f^3$; since $R_0 = R_f$,

$$V \approx 2\pi R_f^2 h_f \qquad h_f = \tfrac{1}{2}h_0 \qquad (14.1b)$$

(Note that a material balance is not required since the densities of the starting "preform" and the resulting thermoformed object are the same.)

For "all" other molds, a volume balance yields the following ordinary differential equation:

$$-\frac{dh}{dz} = \frac{Ah}{B - Cz} \qquad (14.2)$$

as shown by Garrett[70] and subsequent researchers. The various shapes studied so far have been recently classified by Gerstle[71] as "symmetric" molds in which there is a single geometry of the film frozen each instant, versus "asymmetric" molds in which there are two different geometries of film frozen each instant. In addition, there are "one-plane" molds, which are defined by a single line (either extended in one direction or rotated around a given axis), versus "two-plane" molds, which require two such lines. The following outline includes references to the cited works:

- Symmetric Molds
  - **(a)** One-plane molds: Hemispherical,[71,72] open cylindrical,[71,72] conical,[70,71,73,74] triangular[70,71,73,74]
  - **(b)** Two-plane molds: Deep truncated triangular,[70,71,73,74] shallow truncated triangular[70,73,74]

- Asymmetric Two-Plane Molds: Deep truncated conical,[74] shallow truncated conical,[74] deep cylindrical,[71,72] shallow cylindrical[71,72]

Garrett[70] initiated these studies by developing a Galerkin finite-element program in FORTRAN. Wooldridge[74] rewrote it for Apple II computers and added new geometries; she also corrected the solution of Rosenwieg[73] for the shallow truncated cone. Passifiume[72] used FORTRAN and added new geometries; she also demonstrated the need to redefine the finite-element mesh for positions near the "end" of the process. Herps[75] tackled the problem of "edges"—locations where two planes meet; she solved the biharmonic and von Karman equations for an hemioctahedral mold. Gerstle[71] developed a spreadsheet model for thermoforming, utilizing the Runge–Kutta–Fehlberg fifth-order integration scheme in Visual Basic.

The open cylindrical mold is described by

$$-\frac{dh}{dz} = \frac{h}{R} \tag{14.3a}$$

since $C = 0$; "open" refers to the fact that this hole has no bottom (i.e., the mold is *very* deep). The nonuniform wall thickness is given by the analytical solution

$$h_f = h_i \exp\left(-\frac{2z}{D}\right) \tag{14.3b}$$

as shown by Passafiume;[72] here, the initial touch occurs when R just equals $\frac{1}{2}D$, where D is the diameter of the hole.

The conical mold is described by Eq. 14.2 with

$$A = \frac{\sin^2 \beta \tan \beta}{1 - \cos \beta} - 2 \qquad B = H \qquad C = \sin \beta \tag{14.4a}$$

where β is the angle of the cone measured from the horizontal to the side of the mold and H is the axial height of the cone; the analytical solution for the nonuniform wall thickness is

$$h_f = h_i \left[1 - \left(\frac{z}{H}\right) \sin \beta\right]^{\sec \beta - 1} \tag{14.4b}$$

where

$$h_i = \tfrac{1}{2}h_0(1 + \cos \beta) \tag{14.4c}$$

The triangular mold is described by Eq. 14.2 with

$$A = \frac{1}{\beta} - \frac{1}{\tan \beta} \qquad B = \frac{H}{\sin \beta \tan \beta} \qquad C = \frac{1}{\tan \beta} \tag{14.5a}$$

where β is the angle of the mold measured from the horizontal to its side and

H is the center-plane height of the mold; the analytical solution for the nonuniform wall thickness is

$$h_f = h_i\left[1 - \left(\frac{z}{H}\right)\sin\beta\right]^{(\tan\beta)/\beta-1} \tag{14.5b}$$

where

$$h_i = \left(\frac{h_0}{\beta}\right)\sin\beta \tag{14.5c}$$

The deep truncated triangular mold is defined by two converging inclined sides, each forming an angle β with the horizontal, truncated by a third horizontal plane; the adjective *deep* refers to the fact that the initial touch of the free film with the mold takes place on the sides, rather than the bottom. This mold is symmetric in that the linear strip laid down on the bottom after it is touched has the same geometry as the linear strip laid down on the sides. Prior to the bottom being contacted, the sides of this mold have the nonuniform thickness given by Eq. 14.5b.

After the bottom is touched there are four linear strips being frozen, one on each side and two on the bottom, each of these moving away from the centerline of that bottom. Hence, there is contiguity but lack of mathematical continuity in the nonuniform wall thickness distribution; i.e., the slope of the curve that relates h to z changes when the bottom is contacted. The bottom is touched at the instant

$$z_t = \frac{H(1 + \cos\beta)}{\sin\beta} - \frac{W}{2} \tag{14.6a}$$

where W is the width of the mold at the top. Now,

$$A = \frac{2}{\beta} - \frac{1}{\tan(\beta/2)} \qquad B = \frac{H}{\sin(\beta/2)\tan(\beta/2)]} \qquad C = \frac{1}{\tan(\beta/2)} \tag{14.6b}$$

and

$$h_f = h_t\left(\frac{H/\sin\beta - z}{\frac{1}{2}W - H/\tan\beta)}\right)^{[2\tan(\beta/2)]/\beta-1} \tag{14.6c}$$

where h_t is the h of Eq. 14.5b when z is the z_t of Eq. 14.6a.

The shallow truncated triangular mold is defined the same as the deep, but the adjective *shallow* indicates that the bottom is touched by the free film before the sides. Using x to indicate the distance outward from the centerline towards the side, the sides are contacted when

$$x_t = \frac{W}{2} - \frac{H(1 + \cos \beta)}{\sin \beta} \tag{14.7a}$$

For $x < x_t$,

$$h = \frac{h_i R_i \theta_i}{R \theta F} \tag{14.7b}$$

where

$$h_i = \frac{W h_0}{2 R_i \theta_i} \tag{14.7c}$$

$$R = \frac{(\frac{1}{2}W - x)^2}{2H} + \frac{H}{2} \qquad R_i = \frac{W^2}{8H} + \frac{H}{2} \tag{14.7d}$$

$$\theta = \arctan\left(\frac{\frac{1}{2}W - x}{R - H}\right) \qquad \theta_i = \arctan\left(\frac{\frac{1}{2}W}{R_i - H}\right) \tag{14.7e}$$

$$F = \exp\left(\int_0^x \frac{dx}{R\theta}\right) \tag{14.7f}$$

The deep truncated conical mold is defined by one plane rotated around an axis, forming an angle β with the horizontal, truncated by a second horizontal plane; the adjective *deep* refers to the fact that the initial touch of the free film with the mold takes place on the side rather than the bottom. This mold is asymmetric in that the circular strip laid down on the bottom after it is touched has a different geometry than that laid down on the sides. Prior to the bottom being contacted, the sides of this mold have the nonuniform thickness given by Eq. 14.4b.

After the bottom is touched, there are two circular strips being frozen, one on the lateral side and one on the bottom; the latter is moving away from the center point of the bottom. Hence, there is contiguity, but lack of mathematical continuity, in the nonuniform wall thickness distribution; i.e., the slope of the curve that relates h to z changes when the bottom is contacted. The bottom is touched at the instant

$$z_t = \frac{H(1 + \cos \beta)}{\sin \beta} - \frac{W}{2} \tag{14.8}$$

where W is the diameter of the mold at the top. (The equations for determining $h(z)$ after the bottom is touched are complicated; see Wooldridge.[74])

The shallow truncated conical mold is defined the same as the deep, but the adjective *shallow* indicates that the bottom is touched by the free film before the side. Using x to indicate the distance outward from the center point towards the side, the side is contacted when

$$x_t = \frac{1}{2} W - \frac{H(1 + \cos \beta)}{\sin \beta} \tag{14.9}$$

[The equations for determining $h(z)$ after the side is touched are complicated; see Wooldridge.[74]]

The deep cylindrical mold is defined as a truncated open cylindrical mold, where the side is contacted before the bottom; hence, prior to the bottom being touched, the nonuniform wall thickness is given by Eq. 14.3b. After this, the thickness is given by the solution to Eq. 14.2 with

$$\begin{aligned} A &= (\pi - 2)R - (2\pi - 6)z \\ B &= 2R^2 \\ C &= (\pi - 2)(R - z) - 2R \end{aligned} \tag{14.10}$$

The shallow cylindrical mold is defined as a truncated open cylindrical mold, where the bottom is contacted before the side is touched; hence, prior to the side being touched, the nonuniform wall thickness is given by Eq. 14.2 with

$$\begin{aligned} A &= [(H - R)(H + R + 2z) - 3z^2] \operatorname{arccot} \left(\frac{R - z}{H} \right) \\ B &= R(H^2 - R^2 - z^2) \\ C &= \frac{B}{R} \end{aligned} \tag{14.11}$$

After this, Eq. 14.10 is applicable.

15 ROTATIONAL MOLDING

Rotational molding is used to make relatively thick-walled hollow objects, where only the outside of the part has a well-defined geometry; the inside surface depends on the effectiveness of the spinning process.

An appropriate weight of polymer pellets are placed within the two mold halves; this weight is approximately the product of the surface area, the required part thickness, and the density of the solid polymer. The mold is closed, heated, and rotated on two axes. When sufficient time has elapsed for melting to take place (determined by solution of the heat transfer problem), the mold is cooled, the polymer solidified (another heat transfer problem), and the mold is opened to remove the part.

A 55-gal barrel, with an integral lid, may be manufactured this way; the solid that bridges the lid to the barrel is cut, allowing the lid to be separated and subsequently snapped onto the barrel.

PART V. Cyclic Process: Quiescent

16 CASTING

Casting is a quiescent process used to encapsulate nonpolymer objects. This is often used for *objets d'art,* such as water-white paperweights with butterflies, coins, etc., enclosed within. The industrial object studied by Ramanathan[66] was the epoxy encapsulation of small transformers, such that the electrical contacts were exposed. He found that this process, which lasted about 30 min, could be monitored by dynamic dielectrometry. This method followed the resistance, capacitance, and dissipation function of the epoxy resin as it cured, utilizing a 120-Hz AC source. This technique may be used to increase the rate of production by replacing fixed time schedules such as "cure for *t* minutes," with curing times based on objectively measured data.

17 CONCLUSION

Nearly one-half of this brief discourse on plastic parts processing presented the underlying principles. The remaining summaries of the individual processes are, at best, "introductions"; each has had several entire books written on them. One should consult the following Bibliography for sources of further information.

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CHAPTER 34

COMPOSITES FABRICATION PROCESSES

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1 INTRODUCTION

1.1 Nature of Composites

Composite materials (see Chapter 12) are composed of a continuous phase, or matrix, with a discontinuous reinforcement phase dispersed within the matrix. The matrix may be polymeric, metallic, or ceramic and the reinforcement may, likewise, be polymeric, metallic, or ceramic. The discussion in this chapter will, however, be limited to polymeric matrices and the reinforcements will be glass, carbon (graphite), or the high modulus textile fibers based on poly-aramids and polyolefins. The reinforcing phase normally consists of fibers, although some particulate reinforcement is used. There is a subtle distinction between *particulate composites* in which the particles *reinforce* and *filled plastics* where the particles are added for reasons other than stiffness and strength enhancement. The fibers are typically of the order of 10 μm in diameter (about half that of a human hair), but some are less than 5 μm and others up to 150 μm. The fibers may be *continuous* (i.e., dimensions that could stretch right across a workpiece, up to several meters), *long discontinuous* [which are defined here as having an aspect ratio (length/diameter) greater than 10,000 (length of a few centimeters)], and *short discontinuous,* which have an aspect ratio less than 1000 down to 2 (lengths of a few millimeters or less). The fibers may be arranged within the matrix in various ways, which are described as uniaxial or longitudinal (i.e., all fibers aligned in one direction), biaxial in plane, multiaxial in plane, random in plane, and three-dimensional arrays which may be organized (e.g., orthogonal) or random. The properties of the fibers and their disposition in the matrix control the mechanical properties of the composite and also profoundly influence the ways in which they may be processed.

1.2 Role of Fiber Architecture on Mechanical Properties

Fiber Properties

The fibers used in composites have been developed to have very high strength, very high stiffness, or both relative to the polymeric matrix. They mostly also have low density compared with metals and specific mechanical properties exceeding those of most metallic alloys. A comparison of some properties of the principal fibers used as reinforcements with those of typical engineering alloys is given in Table 1. The specific stiffness (E/ρ) and specific strengths (σ/ρ) have been normalized to those of mild steel. These show that the fibers have a stiffness advantage over metals of up to 6.5 times and strength advantage of over 100 times in some cases. It should be noted that the properties of a composite will generally be less than half that of the fiber depending on the proportion of fiber and fiber architecture. This still results in some composites showing a significant advantage over metals.

Fiber Architecture

There are two related aspects of fiber architecture; the fiber volume fraction, V_f, and the fiber orientation. A perfectly collimated uniaxial array of fibers may be packed to a V_f of 0.9, i.e., 90% of the volume is filled by fibers. However, the practical maximum is closer to 0.7. Such an array would result in a composite with stiffness in the fiber direction of approximately 70% of that of the fibers. The transverse properties would be much lower and closer to those of the matrix. On the other hand if the fibers were arranged in a random but planar array, the maximum achievable V_f would be less than 0.3; and, since the fibers are at random, the stiffness would be uniform in all directions, but only about 10% of

Table 1 Comparative Properties of Reinforcement Fibers and Typical Metal Alloys

Material	E (GPa)	σ (GPa)	ρ (kg/m^3)	E_{SR}	σ_{SR}
E-glass fiber (typical)	70	3.5	2540	1.1	54
Aramid fiber (Kevlar 49)	127	2.6	1470	3.4	69
Polyethylene fiber (Spectra 1000)	100	3.0	970	4.0	121
High-strength carbon fiber (T300)	230	3.6	1800	5.0	78
Intermediate modulus carbon fiber (T800)	300	5.8	1800	6.5	126
Mild steel (typical)	200	0.2	7800	1.0	1.0
Alloy steel (typical)	200	1.0	7800	1.0	5.0
Aluminum alloy (7000 series)	72	0.8	2700	1.0	12

E, Young's modulus; σ, maximum tensile strength; ρ, density; E_{SR}, normalized specific stiffness; σ_{SR}, normalized specific strength.

that of the fiber. The mechanical properties are completely dominated by these two considerations and, thus, control of fiber architecture is a vital consideration when selecting a processing route.

Fibers for reinforcement are available either as continuous *tows* or *rovings* consisting of parallel, untwisted arrays of 100 to more than 50,000 filaments, or in various sheet or web forms suitable for making laminates. The most common sheet formats are random mats, woven or knitted fabrics, braids and nonwoven, stitch-bonded fabrics. These are discussed briefly in the next section.

Commercial Reinforcement Formats

Tows are bundles of fibers containing from about 100 to more than 10,000 fibers. These bundles are generally untwisted. Twisted forms are designated *yarns,* although these terms are often confused. Rovings are formed by combining a number of tows to form a larger bundle, as shown in Fig. 1. These continuous bundles are supplied in spools or ‘‘cheeses,’’ often containing up to 100 kg of fiber. The size of the bundle is usually designated by the mass of a standard length. The international standard is the *tex,* which is the mass in grams per kilometer of yarn. An alternative, still used in the United States, is the *denier,* where 1 tex = 9 deniers. Rovings used for reinforcement typically have linear masses ranging from 50 to 5000 tex.

Random mats may be manufactured in two ways. The first is formed by chopping rovings into short lengths, typically 10–75 mm, and letting these fall freely onto a moving belt. The resulting random planar array of short bundles is consolidated by application of a binder and passing between rolls. The resultant roll of mat is termed *chopped strand mat* or CSM. Areal weights of CSM are generally in the range of 100–2500 g/m^2. The second method is to form a mat by placing random swirls of continuous roving onto a moving belt and then consolidating in a similar manner to that of CSM. The product is known as continuous *random mat,* CRM, or *swirl mat*. It is produced in similar areal weights as CSM.

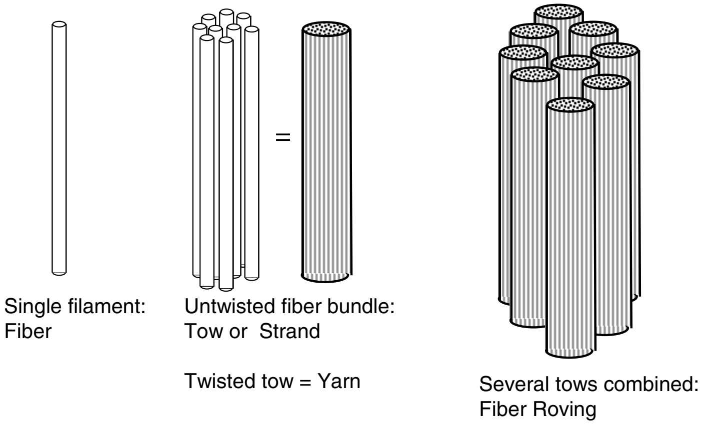
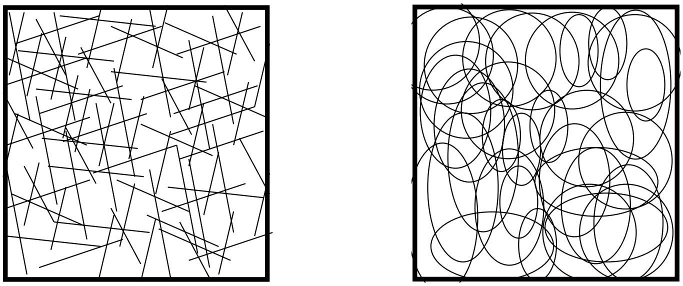


Fig. 1 Schematic illustration of single filaments, tows, and strands and rovings. Note that the term “yarn” strictly refers to *twisted* tows but is often used interchangeably.

These two products are very widely used in the industry. They allow a maximum V_f of about 0.3 to be achieved with a random planar fiber orientation distribution. They are cheap and easy to process. Their molding qualities depend on the areal weight of the mat and the type of binder employed. The structures of CSM and CRM are shown schematically in Fig. 2.

Woven fabrics are manufactured using standard textile weaving technology from fiber rovings and to some extent yarns. Standard fabrics are biaxial, i.e., they have weft (fill) and warp rovings set at right angles. Plain weaves, twills, and satin fabrics are commonly used at areal weights between 200 and 5000 g/m^2. Special fabrics include planar triaxial formats, three sets of rovings at 120° to each other, and three-dimensional forms. Two important aspects of woven materials are the degree of crimp and drapeability. Crimp is the degree to which the rovings deviate from a linear path as they pass over and under each other. Crimp is undesirable as it reduces the reinforcement efficiency. Satin weaves have less crimp than plain weaves, as shown in Figs. 3*a* and 3*b*. The other aspect is drape, which is the degree to which the fabric is able to conform to contours on the mold. Lightweight cloths and satin weaves generally show better drape than plain weaves. Any binder incorporated into the reinforcement will also influence drape. Braid may also be considered as a woven form of reinforcement. Braid is typically tubular and is formed by counter rotating braiding wheels that carry the roving packages. Braids are usually manufactured with weaves designed for specific products. They are particularly suited to hollow tubular forms and many variations in structure are possible. Knitted fabrics are also manufactured for reinforcement, these may be either flat or tubular as knitted. The general feature is a high degree of crimp and low fiber fraction. However, it is possible to *fill* the knitted form with heavy unidirectional rovings aligned in one or more directions (cf. noncrimp fabrics below), so that the knitted structure holds together the main reinforcement.

Noncrimp fabrics, as the name implies, have been developed to eliminate the problem of crimp. Unidirectional arrays of rovings are bonded together by stitching using a light thread, usually polyester, sometimes aramid. Several layers may



Chopped strand mat - CSM
Randomly oriented bundles
of fibers - 25 - 75 mm long

Continuous random mat - CRM
Random swirls of continuous
fiber tows

Fig. 2 Illustration of the structure of chopped strand mat (CSM) and continuous random mat (CRM), also termed *swirl mat*.

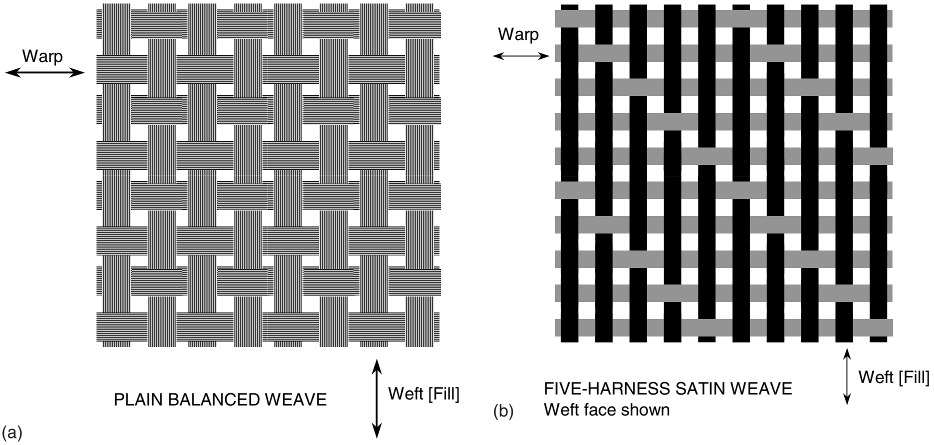


Fig. 3 (*a*) Structure of a balanced plain woven cloth. The warp and fill tows cross in an alternating over and under sequence. (*b*) A 5-harness satin weave. The fill tows pass under one, then over four warp tows in a repeated sequence. This results in less crimp and better drape characteristics than a plain weave.

be bonded together with the fibers aligned differently in each layer. Thus, one layer of noncrimp fabric is equivalent to up to four, sometimes more, uniaxial plies. Typical orientations are 0°, 90°; 0°, ±45°, 90°; and 0°, ±45°. Areal weights range from about 400 to 3000 g/m^2. The rovings in each layer can be very closely packed so that the fabric results in composites with higher V_f than can be achieved with woven material. It is also possible to incorporate layers of mat, woven, or core-forming material between the plies. A further possibility is that the construction of the fabric can be modified by a carefully designed set of open channels within the layers to increase the permeability allowing faster molding than with equivalent reinforcements.

Precompounded Reinforcements with Thermosetting Matrices

The reinforcements discussed above are *dry,* i.e., they contain no resin, apart from small amounts of binder. The resin matrix has then to be infiltrated into the reinforcement later in the processing cycle. An alternative is to use a precompounded reinforcement, which contains all the resin necessary for further processing. The advantages are that the infiltration is effected in a highly controlled manner that guarantees the correct resin fraction and uniform distribution. The disadvantages are increased cost, restricted shelf life, and more limited processing possibilities. In the first place only certain thermosetting resin are suitable. They need to be in soft but not liquid or too sticky state. They need to be fully formulated, i.e., already contain any necessary curing agents, initiators, or accelerators. This means that the resin must be inherently unstable and will start to cure as soon as the compound is manufactured. The rate of cure is temperature dependent, and the resins used are usually formulated to cure at 100–200°C in 10–60 min. This then allows them to be stored at much lower temperatures between manufacture and final processing without unacceptable advance of the cure. Most commercial systems have a shelf life at room temperature (20°C) of at least one month. However, many require them to be stored at subzero temperatures, typically −15°C, when they have a storage life of up to one year. This complicates the logistics of processing and inevitably adds to the costs.

Compounded reinforcements are available as impregnated tows or rovings, as preimpregnated warp sheet, or *prepreg,* preimpregnated woven fabrics and mats, and preimpregnated random fiber compounds such as sheet molding compound (SMC) and bulk molding compound (BMC). The processing principles are common. The material contains all the fiber and resin (possibly an excess) for manufacture of the component. After assembly by laying tows or laminating sheet forms, the material is subjected to heat and pressure. First the resin softens, allowing consolidation, elimination of trapped gases, and any resin excess, then the resin cures to form the solid part. In some cases, e.g., prepreg, which is comprised of continuous fibers, no lateral flow of the fibers can occur. So the plies have to be precisely tailored to the shape of the part. In other processes, e.g., SMC, there is considerable flow of both resin and fiber so that complex shapes including stiffening ribs and other features may be formed from blanks of simple shapes. Prepreg consists of a uniaxial array of continuous fibers produced as a continuous roll up to about 2 m in width. The resin is applied to the web of fiber and the compounded material then consolidated between two rolls of protective film. One is usually a silicone-coated paper and the other a polyolefin film. The finished standard prepreg has an areal density of 100–200 g/m^2 and when cured is designed to have a thickness of 0.125 mm per ply. Thus 8 plies are required to form a laminate 1 mm thick. While this would be inconvenient for the manufacture of thick structures, it does allow very precise tailoring of the laminate properties by varying the lay-up sequence. A quasi-isotropic laminate may be formed by the 8-ply sequence $[0°, \pm 45°, 90°]_s$, the subscript "s" indicating symmetric. Alternative sequences, usually symmetric, give a different balance of longitudinal, transverse and in-plane shear properties. Prepreg is also manufactured in thinner formats and also using fine woven fabrics of plain or satin weave which typically laminate to 0.25 mm/ply.

There has recently been much interest in developing prepreg-like materials more suitable for manufacturing thick laminates. These are based on combinations of woven and noncrimp fabrics and mats, impregnated with suitable thermosetting resins, usually vinyl-ester or epoxies. Areal densities exceeding 6000 g/m^2 have been produced. The same problems of handling and shelf life apply but, outside the aerospace industry, subzero storage is seldom acceptable and shelf lives exceeding 3 months are demanded. This limits the range of resin systems but satisfactory systems are now available. A concern with all precompounded materials is the degree of *tack* or stickiness. Too little tack means that lamina do not stay in place as the laminate is laid up. Too much tack renders the material difficult to handle as it sticks to everything it contacts. Aerospace prepregs are manufactured to a precisely controlled tack, but even this tends to lessen as the material approaches the end of its shelf life. A technique recently introduced for heavier reinforcements is to make a multiple layer material but to confine the resin impregnation to the central layers, shown in Fig. 4. Such a material handles like a *dry* reinforcement, although it tends to be much less *springy*; when subjected to heat and pressure, the resin content softens to a low viscosity and flows uniformly throughout the reinforcement. Developments such

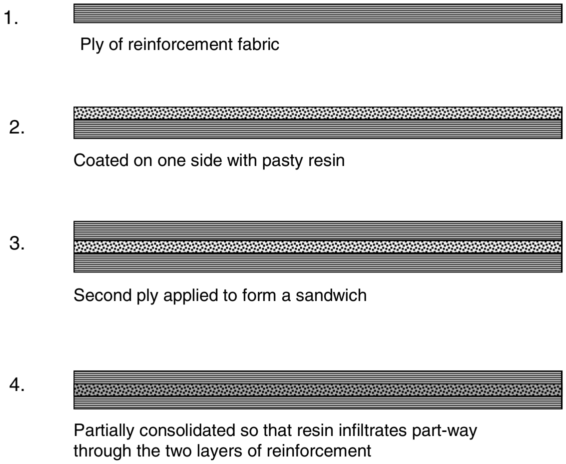


Fig. 4 Sequence of manufacture of a partially impregnated fabric reinforcement. A layer of pasty resin is sandwiched between two layers of fabric or mat. This produces a dry material with good handling characteristics and is particularly suitable for heavier grades of reinforcement.

as this illustrate how the industry is using imaginative techniques to improve productivity while maintaining quality.

Sheet molding compound (SMC) and bulk molding compound (BMC) are well-established precompounded materials, which are widely used in the automotive and general engineering industries. SMC is manufactured by impregnating a continuous thick mat of chopped glass rovings, typically 50–75 mm long, with a suitable resin, usually an unsaturated polyester or vinyl-ester containing a large proportion of particulate fillers, such as chalk. The resin is specially formulated so that it hardens to a thick paste on maturation after the compounding operation. This takes about 72 h. The product is a continuous sheet of resin-impregnated chopped rovings, typically about 5 mm thick and up to 2 m wide. It has a dry but pliable consistency with no tack. It may be cut using shears or a blanking tool. The cut pieces are compressed between hot matched tools. At first the resin softens allowing the charge to flow to fill the mold; then the resin cures. The part is ejected hot. The whole process takes from 5 to 20 min, according to the size and complexity of the molding. This material and process is particularly compatible with the production environment in the automobile and other mass production industries. BMC is of similar formulation but typically uses shorter fiber bundles, 10–25 mm, and is formed into a *rope,* ~50 mm diameter. This is used as feedstock for either compression or injection molding. Once again the parts are ejected hot and the molding cycle can be as short as 1 min. Both materials consist of flow-oriented quasi-random arrays of fiber bundles and the fillers in the cured resin matrix.

Thermoplastic Matrices

These present a totally different set of processing considerations. Thermoplastics are processed by a melt–freeze cycle and no chemical curing reactions are involved. The materials are always solids at ambient temperature, and common systems melt at temperatures ranging from 150 to 400°C. The main processing problem with thermoplastics is that their viscosity in the molten state is orders of magnitude higher than that of the common thermoset systems prior to curing. There are a few thermoplastics, e.g., polyamides 6, 11, and 12, that can be processed by *in situ* polymerization of monomer precursors and are exceptions to this generalization. Preimpregnated tow and sheet materials comparable with prepreg and SMC can be prepared, but they are rigid and boardlike below their melting point. They are processed by preheating and then pressing or stamping between cold tools. In general thermoplastic-based systems are limited to somewhat lower V_f than comparable thermoset composites. An alternative strategy is to prepare commingled products by combining reinforcement fibers with thermoplastic fibers by spinning or weaving together. An extended discussion of these products will be found in Section 15.

Mechanical Properties of Laminates and Moldings

As indicated earlier, the mechanical properties of a composite are controlled by the proportion of fiber, V_f, and the overall fiber architecture or fiber orientation distribution. Fully uniaxial laminates have the highest stiffness and strength in the fiber direction but are relatively weak in the transverse direction and in shear. They are seldom used because they can split parallel and between the fibers. Most practical laminates therefore contain a proportion of their fibers in different directions. A balanced woven fabric has about 50% of its fiber in each of two directions mutually at right angles. A quasi-isotropic laminate has 25% of its fibers in each of the four directions 0°, 90°, and ±45°. In each of these cases the laminate will be less anisotropic than the uniaxial but the actual stiffness and strength will be lower. A lower fiber fraction will reduce the stiffness and strength in all of these directions. A random in-plane distribution, such as results with the use of CSM, gives uniform properties in all in-plane directions but at a level of less than a third that of a uniaxial array. This must be factored by the fact that the V_f will be no more than 0.3 compared with a possible 0.7 for the uniaxial. It is useful to calculate an orientation constant, C_0, for each type of reinforcement; this is 1 for uniaxial, 0.5 for the balanced biaxial fabric, and 0.3 for random in-plane. This may then be multiplied by the actual V_f to give a reinforcement efficiency factor, η_R. Table 2 indicates the reinforcement potential for a number of common forms of reinforcement calculated for an E-glass/epoxy resin system. $E_{\max}$ is the Young's modulus in the stiffest direction and $G_{\max}$ is the in-plane shear modulus, both in units of GPa. Note that the maximum achievable V_f decreases as the format becomes more random and also that the inclusion of the ±45 orientation enhances the shear modulus. As V_f decreases, the values for E and G converge toward those of the resin. The symbols RTM and RFI are for resin transfer molding and resin film infusion, respectively.

2 BASIC PRINCIPLES FOR PROCESSING

2.1 Aims and Objectives

The aim in selecting a processing route is to manufacture the part to meet all the specified objectives at the required production rate and at the lowest possible cost. Selection of a processing route certainly includes selection of appropriate

Table 2 Implications of Choice of Reinforcement Format for E-glass/Epoxy Resin

Reinforcement	Format	V_f max	C_0	η_R	E_{max} (GPa)	G_{max} (GPa)	Applicable Processes
Uniaxial tow	0°	0.80	1.00	0.80	56	5.0	Tow placement, filament wind, pultrusion
UD prepreg	0°	0.65	1.00	0.65	47	5.0	Autoclave, RFI
Biaxial prepreg	0°, 90°	0.60	0.50	0.30	22	5.0	Autoclave, RFI
Quasi-isotropic prepeg	0°, ±45°, 90°	0.60	0.33	0.18	13	8.0	Autoclave, RFI
Balanced woven	0°, 90°	0.50	0.50	0.25	18	4.1	RFI, RTM, contact mold
Isotropic woven	0°, 90°; ±45°	0.50	0.33	0.16	12	7.0	RFI, RTM, contact mold
Noncrimp fabric	0°, 90°; ±45°	0.55	0.33	0.18	13	7.5	RFI, RTM, contact mold
Orthogonal 3D	x, y, z	0.40	0.33	0.13	9.4	4.0	RTM
CSM	Random 2D	0.30	0.33	0.10	7.2	3.5	RFI, RTM, contact mold
CRM	Random 2D	0.20	0.33	0.07	5.0	3.0	RFI, RTM, contact mold
Short fiber	Random 3D	0.15	0.12	0.02	3.4	3.0	Injection mold
Unfilled epoxy resin					0.3	2.5	

feedstock and may also involve selection of the basic material system, e.g., E-glass/polyester or carbon/epoxy.

2.2 Common Features of Composite Processing Routes

Infiltration

The reinforcing fibers are initially produced as spools of continuous roving. This may be treated with a *size* and/or binder but is otherwise dry. During the processing operations, it must be fully infiltrated with the matrix resin. This may be accomplished either by some precompounding operation or during the final molding process. Infiltration of a porous medium by a liquid has been extensively studied and a simple relationship proposed by Darcy is widely used:

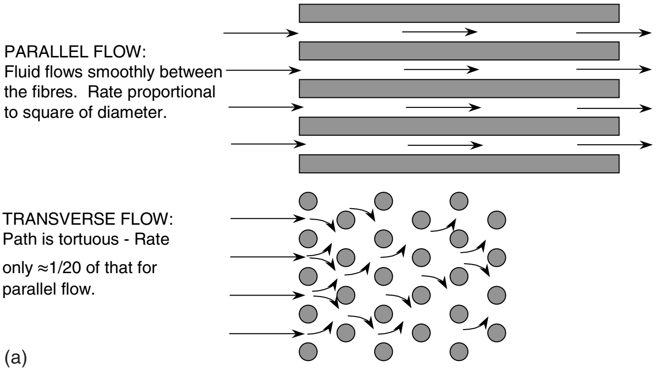
$$q = \frac{S\,\Delta p}{\eta\,\Delta L} \tag{1}$$

The rate of infiltration, q ,is proportional to the permeability, S, and the driving pressure gradient, $\Delta p/\Delta L$, and inversely proportional to the viscosity of the medium, η. Thus, increasing the applied pressure or the permeability will assist infiltration while increasing viscosity will hinder. Viscosity may usually be decreased by raising the temperature, but this leads to faster cure in thermosets and ultimately to degradation in thermoplastics. It is necessary to establish a *processing window* where the temperature and pressure are controlled to allow complete infiltration to be effected before gelation or degradation of the matrix resin. The length L over which infiltration must occur is basically determined by the geometry of the part. However, the effective infiltration path can be altered by multiple gating and also in rather more subtle ways by design of the reinforcement.

The permeability, S, is determined by the reinforcement architecture and may be modeled using the Carman Kozeny relationship:

$$S = \frac{r_f^2(1 - V_f)^3}{4kV_f^2} \tag{2}$$

Here the permeability of a uniform array (of fibers) is given by the square of the fiber radius, a function of V_f, and the Kozeny constant, k. It can be seen that the permeability will be lower when the fibers are of smaller radius. The effect of V_f is very severe, and the permeability decreases by a factor of about 6 for a V_f increase from 0.6 to 0.7. The Kozeny constant is a measure of tortuosity: In a uniform uniaxial array of cylindrical fibers (Fig 5*a*), k for flow parallel to the fibers is some 20 times lower than that for flow normal to the fibers. This



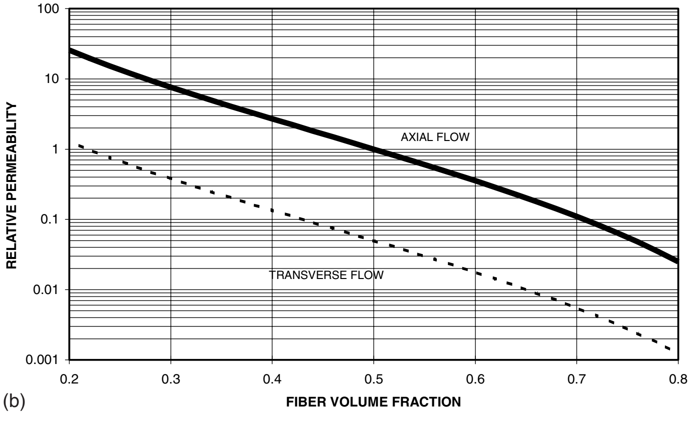


Fig. 5 (*a*) This depicts flow parallel and normal to a uniaxial array of fibers. The tortuous flow in the normal direction results in a flowrate only $\approx\frac{1}{10}$ of that in the parallel direction under similar conditions. (*b*) Relative permeabilities in the axial (parallel) and transverse (normal) directions of uniaxial fiber arrays is plotted against fiber fraction, V_f. Note that the transverse flowrate is only $\frac{1}{20}$ of that in the axial direction. There is a reduction of permeability by a factor of approximately 100 for an increase in V_f from 0.3 to 0.7.

effect and that of V_f are further illustrated in Fig. 5*b*, which shows that permeability is reduced by a factor of 10 for an increase in V_f from 0.4 to 0.6. These two relationships allow some simple generalizations to be stated:

(i) Permeability will be higher for a coarser fiber array.
(ii) Permeability is much higher in the fiber direction than at right angles.
(iii) Permeability decreases sharply as V_f is increased.

Unfortunately small-diameter fibers at high V_f tend to give composites with better mechanical properties, so it is necessary to balance the aims of achieving high mechanical properties against the requirements of ease of processing. Fortunately there are a number of ways in which the effective permeability may be increased without undue performance penalty. These are discussed in a later section.

Consolidation

This is the process whereby the thickness of the section is reduced to the design value and porosity and other defects eliminated. When dry or impregnated plies (or tows) are laid up to form a laminate, the initial lay-up (pack) will be significantly thicker than ultimately required. This is due to the difficulty in obtaining perfect contact, to entrapment of air, and sometimes due to an excess of resin that is often deliberately incorporated. The application of pressure and/or heating to the pack will cause it to consolidate. Ideally, any entrapped air or other volatiles will also be eliminated together with any excess resin. Provision for removal of resin and volatiles may be made by venting the tooling, by the use of absorbent bleeder fabrics, and/or by the use of vacuum. The consolidation process requires that resin flows, usually in the thickness direction of the pack. The process may be partially affected by a preconsolidation operation (debulking) prior to the main processing operation or as part of the main process.

The principles of consolidation of a simple laminate, with a bleed ply, are illustrated in Fig 6. Once again the process requires a sufficient time for the flow processes to occur and this will be affected by the applied pressure and the temperature. Where preconsolidation is used, care must be taken not to advance the cure of thermoset systems too far so as to compromise satisfactory final consolidation and cure.

Effects of Applied Pressure

Examination of the Darcy relationship (Eq. 1) indicates that the rate of infiltration may be increased by increasing the pressure gradient forcing infiltration. While this is true, there are some limitations and implications that must be discussed. First, much composite molding is carried out using simple tooling, usually also composite, and a vacuum bag. This limits the pressure that may be applied to less than one atmosphere ($\approx$1 bar). Higher pressures may be applied if an autoclave is employed, typically up to 5 bars. The limitation here is the pressure capacity of the autoclave vessel, which may be 3 m in diameter. Higher pressures may be exerted by press, but it should be noted that to apply a pressure of only 10 bars, which is approximately 1 MPa, a closing force of 100 ton·f is

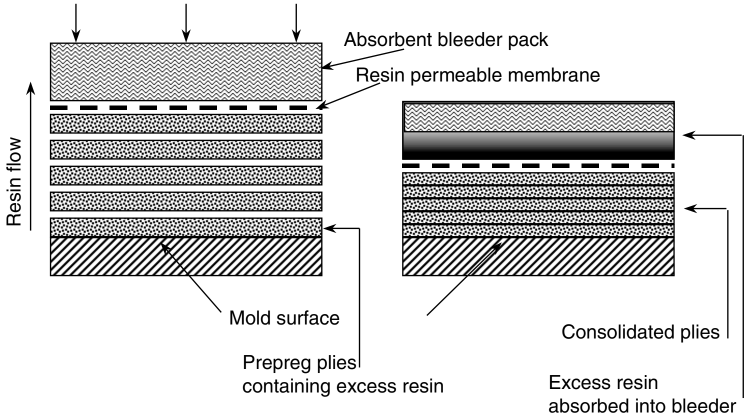


Fig. 6 Process of consolidation involves flow in the thickness direction of the laminate. If a bleeder ply is used, excess resin flows through a resin-permeable release ply and is absorbed in the porous bleeder.

required for each 1 m^2 projected area of the molding. One implication of using higher pressures is that the tooling needs to be made more robust. This means use of steel tools, at a cost of at least five times that of the simple composite tooling. A further problem is that the application of high compressing pressures to the reinforcement before it is infiltrated, will tend to consolidate it, increase the V_f, and decrease the permeability, so that there is no gain in infiltration rate.

Cure of Thermosetting Resins

Thermosetting resins are cured by a chemical process that is enhanced by raising the temperature. The detailed chemistry of this process will not be discussed, but the basic behavior is common to all systems. The resins are usually supplied in liquid form, typically with the consistency of a light lubricating oil. There are usually at least two components, which must be mixed in precise proportions prior to processing. One component is normally the resin and the other may be designated curing agents, initiators, accelerators, or catalysts. (The term *catalyst* is, however, often used loosely in the industry when the ingredients are not technically catalysts!) Once mixed they are fully formulated and the chemical process leading to cure is initiated. Resins may be formulated to cure at ambient temperature (e.g., 15–25°C) or at elevated temperatures (typically 50–250°C) and over time intervals from a few seconds to several hours, even days. Some systems, notably the unsaturated polyester resins, are more versatile in this context than others.

The curing reaction results in the development of a dense network of chemical bonds between the original polymer molecules. On the completion of cure the resin is solid, cannot be melted, and cannot be dissolved in solvents without degradation. Most of the thermosets used in composites cure to hard rigid solids, but it is possible to formulate systems to yield softer or even elastomeric products. The cure process occurs in several stages. First there is often an incubation period when there is little observable change. Then the viscosity is observed to increase, and heat arising from the reaction(s) will be evolved. This usually results in a rise in temperature, referred to as the cure exotherm. A consequence of this temperature rise is that the viscosity of the resin will be decreased, which assists the processes of infiltration and consolidation. The next stage is gelation: At this point the resin ceases to be liquid and transforms to a solid, initially it is rubbery but then becomes progressively harder. The evolution of heat is generally most intense during the period around gelation. As the cure proceeds the exotherm becomes weaker and the resin becomes fully hardened or vitrified. Raising the initial cure temperature by heating will accelerate the cure process. Gelation will occur sooner and the exotherm will be more intense. Care must be exercised to ensure that the exothermic heat does not lead to the resin/laminate becoming excessively hot so that degradation occurs. This is often observed as *burnt* regions in the thicker sections of a molding where heat transfer out of the laminate is less favorable. In extreme cases the molding can completely disintegrate, or even explode, as a result of excess exotherm! It is fortunate that the reinforcement, which does not participate in the reactions, acts to some degree as an internal heat sink.

Three temperature, viscosity and cure profiles for a laminate cured at different temperatures are shown in Figs. 7*a*, 7*b*, and 7*c*. Note how the time to gelation is reduced by raising the cure temperature. For reasons of productivity it is generally desirable to cure as fast as possible, provided excessive exotherm can be controlled. However, it is also necessary that the resin is at low viscosity for a sufficient time interval to allow any infiltration or consolidation processes to be completed. This may be achieved by balancing the cure temperature profile and the consolidation pressure.

Processing Thermoplastic Matrix Systems

Thermoplastics, with the exception of the monomer polymerization systems, are not cured but are processed through melt–freeze cycles. Thermoplastics are either amorphous or semicrystalline, with degrees of crystallinity of up to about 75%. On heating to above the melting temperature, T_m, amorphous polymers soften over a range of temperatures while the semicrystalline materials melt more sharply and generally to melts of lower viscosity. Overheating leads to degradation, discoloration, and loss of properties. On cooling back through the freezing temperature, the amorphous materials become gradually more rigid, but the semicrystalline polymers freeze more sharply and recrystallize. The extent of crystallinity is dependent on the rate of cooling through the critical region. Fast cooling leads to a fine substructure but lower overall crystallinity, while slow cooling has the converse effect. In most cases optimum mechanical performance of the composite requires that the crystallite size and degree of crystallinity be controlled. It is therefore necessary to exercise control over cooling rates when processing thermoplastic systems. A further property of amorphous thermoplastics is the *glass transition temperature,* T_g. This transition occurs below the melting point and at $T < T_g$ the polymer behaves as a glass. It is hard, often brittle, and does not creep significantly under load. At $T > T_g$ the polymer is *rubbery* and much softer, extendible, and creeps under load. The temperature T_g effectively defines the upper service temperature for amorphous thermoplastics. In the case of the semicrystalline thermoplastics only the amorphous portion is affected by T_g, so the effect is less severe. Semicrystalline polymers therefore have higher useful service temperatures and in the temperature interval between

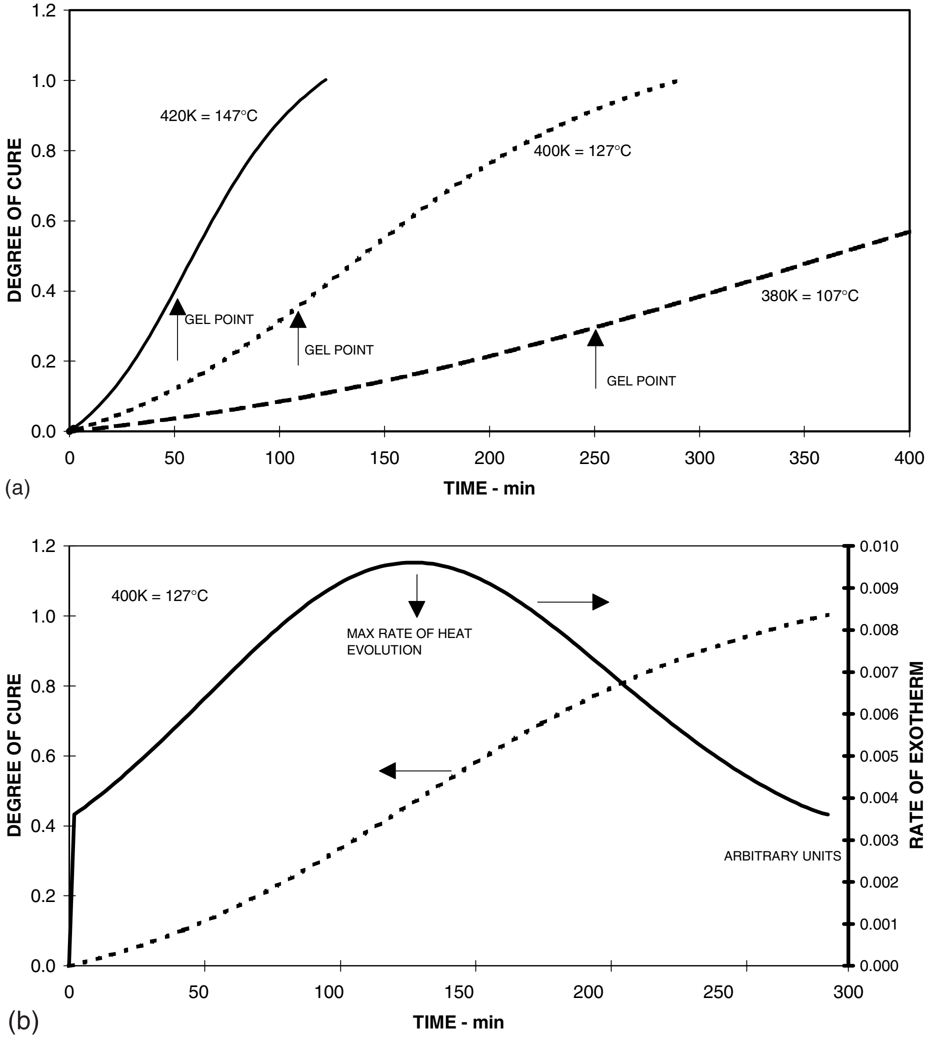


Fig. 7 These figures illustrate the cure characteristics of a typical thermosetting resin, e.g., an epoxy. (*a*) Shows the cure profile at three different curing temperatures. The degree of cure is plotted against time of cure. The cure rate is much faster at the higher temperatures. A temperature increase of only 20°C, from 107°C to 127°C, reduces the gel time from 250 min to just over 100 min. (*b*) Cure profile at 127°C is shown together with the rate of exotherm. This peaks soon after gelation. The exotherm rate is higher at higher cure temperatures (not shown). (*c*) Resin viscosity profiles for the same resin at the three temperatures is shown. The viscosity increases sharply after the gel point and this defines the workable range, or *process window*.

T_g and T_m are often quite tough. The high melt viscosity of thermoplastics means that processes dependent on infiltration cannot be used. Most systems are, therefore, precompounded or co-mingled.

Heat Transfer

It will have become apparent from the preceding discussion that most processing operations involve heat flow, to manage heating, cooling, or to control exotherm or crystallinity. Polymer resins are generally poor thermal conductors and are

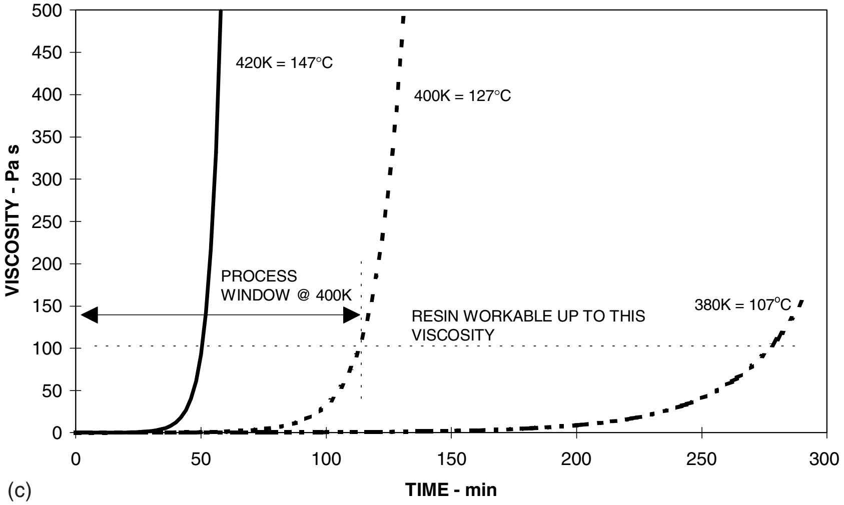


Fig. 7 (*Continued*)

sensitive to overheating. Unimpregnated fiber arrays are also very poor conductors, although some fibers themselves are quite good. The materials used for tooling may also be poor conductors. This applies particularly to composite tooling, which is very widely used, also to plaster and concrete. The problem is often increased by the use of vacuum bags, porous breathers, and bleed plies over the laminate on the tool. Metal tooling is the exception, but this is only generally used for the higher production rate processes. It follows that considerable care is needed to manage the heat input and output during processing and that the time required for heat transfer is a significant and sometimes critical part of most processing operations. Problems may be minimized by avoidance of thick sections and by the incorporation of internal heating or cooling into mold tools.

Flow Processes during Molding

In all processes some flow of resin will occur, but the flow or movement of the reinforcement warrants further consideration. At one extreme there are processes that involve vertical consolidation only with no relative movement of the reinforcement in other directions. The other extreme is when molding with a compound, such as BMC, where the whole charge, resin plus fiber, flows in all directions to fill the mold. In reinforcements incorporating straight continuous fibers, no movement can occur in the fiber direction. Any significant movement normal to the fibers will result in splitting if there is extension or wrinkling if there is compression. On the other hand limited shear deformation is possible. If there is biaxial continuous reinforcement, as in a woven cloth, no stretch can be accommodated in either direction, compression will result in wrinkling but again some shear movement is possible.

Reinforcements containing discontinuous fibers, however, are free to flow to a much greater extent. Sheet molding compound, with chopped roving reinforcement, will flow in three dimensions and allow features such as ribs and bosses to be molded without deliberate placement of reinforcement at these features. Continuous random mat will also flow but to a lesser degree. Clearly it is important to select reinforcement appropriate to the degree of flow necessary to form the part. See the comment in the following section concerning thermoplastic matrix systems.

Drape

This is related to flow but is concerned with the extent to which a reinforcement will comply to a complex molding surface. A uniaxial prepreg can only be successfully draped on single curvature surfaces. Double curvature requires extension or contraction in the lateral direction leading to splits or wrinkles. Double curvature panels can, however, be formed by use of narrow tapes with small gaps or overlaps accommodating the misfit. Biaxial continuous fiber reinforcement, in principle, suffers from a similar limitation but can, in fact, accommodate some draping by tow slippage and shear. Satin and twill weaves have significantly better drape characteristics than plain weaves and lighter fabrics drape more efficiently than heavy. Random mats drape very well, but this is affected by the type of binder used. A strong binder will prevent effective drape. If the binder is resin soluble, draping can be effected after the resin has been applied. With CSM there is a possibility of tearing if the binder is too weak. This leads to depletion of reinforcement in the torn areas. Continuous strand mat is resistant to tearing, it drapes well but cannot flow as much as CSM. Both CSM and CRM are used in precompounded thermoplastic sheet systems. They cannot flow in any way until the matrix has been melted, but then behavior is similar to that in thermosets.

2.3 Design of Reinforcements to Enhance Permeability

Following from the discussion of infiltration in Section 2.2, it would appear that the objectives of obtaining high mechanical properties by maximizing V_f and benefiting from high permeability are mutually exclusive. Tightly packed arrays of small-diameter fibers are highly impermeable, especially in the directions normal to the fiber axes. The effect of V_f on permeability is shown in Fig. 5*b* for axial and transverse infiltration. The time to infiltrate a 250-mm-long uniform array of 10-μm-diameter fibers, at a V_f of 0.5, under a pressure of 2 bars with a resin of 0.1 Pa/s is calculated to be in excess of 200 h, i.e., practically impossible! However if the fibers are arranged as tightly packed tows, of 1 mm diameter and packed with a V_f of 0.7 within the tows, and the tows then packed with a packing factor of 0.7, to give an overall V_f of 0.49 ($\approx$0.5), as shown in Fig. 8, under similar conditions the infiltration time of the array is about 5 s. Furthermore the transverse infiltration of the tows takes only a further 15 s because the distance to infiltrate is only about 0.5 mm (half the tow diameter), so that complete infiltration takes about 20 s. This is a much more realistic prospect. The key to high permeability with high volume fraction is to provide a network of relatively large passages through the reinforcement, between tows, or simply by leaving some gaps between tows. The resin can then quickly infiltrate through these large passages by axial macroinfiltration, and then infiltrate the tows by transverse microinfiltration. These infiltration passages may be incorporated into the reinforcement during the weaving operation.

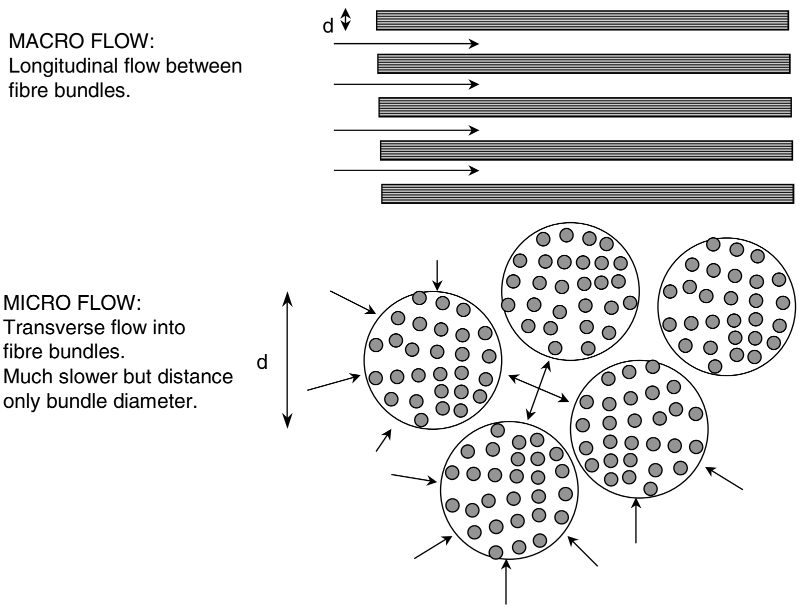


Fig. 8 Illustrates the principles of macro- and microflow in an array of fiber bundles. This is the normal structure of fiber composites. Flow is fast through the relatively large channels between the bundles. Final infiltration is accomplished by microflow in the transverse direction into the tightly packed fiber bundle. This is much slower but the infiltration distance is very short.

Another method of speeding up infiltration is to incorporate layers of open reinforcement, such as CSM, between layers of heavier material such as woven roving or noncrimp fabric. The distributor ply allows the whole area of the molding to be infiltrated so that the heavy reinforcement only has to be infiltrated through its thickness direction. Again the key is to minimize the distance through which tightly packed fibers need to be infiltrated. This principle is further exploited in the SCRIMP and RFI processes discussed in later sections.

2.4 Tooling

Most composite laminates are of shell form, often with additional detail such as stiffening ribs, bosses, and cutouts. These forms may be molded using single-sided tools of either male or female form. The choice is usually governed by the geometry of the piece, the logistics of the laminating process, and which side constitutes the *prime surface* where dimensions and surface finish need to be controlled. Ribs or bosses may be incorporated either by cutting these features into the tool face or, more usually, by building up on the back surface using additional tool pieces where necessary. Simple one-sided tools may be fabricated using composites by taking an impression off a master form, usually termed the *plug*. The basic impression is a thin shell that is then backed up with stiffeners and supporting structure to render it sufficiently robust to withstand the chosen molding process. The mold materials may be E-glass with unsaturated polyester, vinyl ester or epoxy resin matrices, or carbon fiber with epoxy backed up with timber or metallic supporting structures. The advantage of carbon fiber is its greater stiffness, better thermal conductivity, and, most significant, much lower coefficient of thermal expansion. This facilitates molding to closely specified dimensions and is particularly relevant when the molding is also carbon fiber reinforced. In contact molding, laminates are built up on the mold surface and simply hand consolidated without application of additional pressure. Alternatively the mold may be vacuum bagged so that one atmosphere of consolidating pressure may be used. In some instances single-sided tooling may be press consolidated using an elastomeric counter tool, but note that a more robust supporting structure may be necessary. Single-sided tooling is widely used, with a vacuum bag for autoclave cure. Some of these options are illustrated in Figs. 9–11. Double-sided or matched tooling may also be fabricated using composites. This is again usually used in a press, and therefore size and consolidating pressure will be determined by the equipment available.

Composite tooling is suitable for short to medium production runs. Typically a tool may be used to mold up to 100 parts before needing extensive reworking. This figure can, however, vary very widely depending on the complexity of the molding and the consolidating and cure conditions. Where long runs are required, the options are multiple tools or metallic tooling. Metallic tools are inevitably much more costly than composite, but they are more robust and more durable. A serious problem is the mismatch in thermal expansion between the composite part and the tool, which causes difficulty in establishing dimensional control. The options for metal tooling are cast-zinc alloy, cast or fabricated aluminum alloy, cast iron, fabricated steel, and Invar. The choice depends on the part size and complexity, surface finish requirements, and thermal expansion considerations. Invar has the great advantage of virtually zero thermal expansion over typical molding temperature ranges (20–150°C). It is an alloy of nickel and iron and may be fabricated using standard steel practices, the cost is much higher than that of steel. Metal tooling is generally necessary for all high-pressure

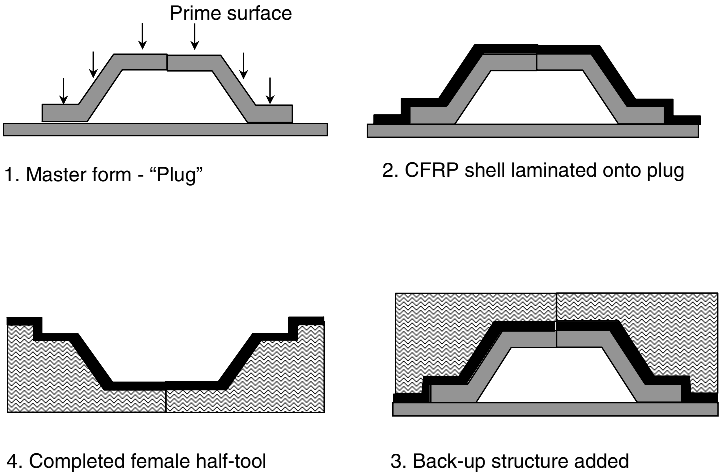
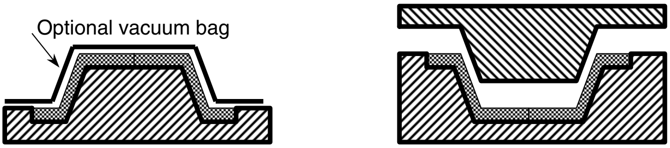


Fig. 9 Composite tool may be manufactured by laminating a CFRP shell onto a master form or plug. This is then backed-up by a suitable structure to provide the necessary robustness for subsequent molding operations.

Female mold

Part laminated into female mold



Part laminated onto male mold

Principle of two-part closed mold

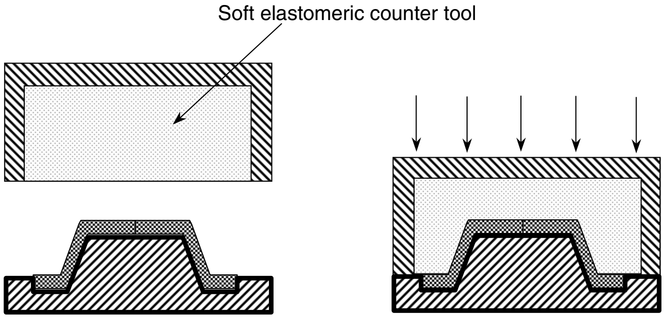
Fig. 10 There are several options for contact molding composite laminates. Single-sided tooling of either male or female form may be used, in conjunction with vacuum bag consolidation, if required. Enhanced shape definition and surface finish may be obtained by using two-part tooling, normally set in a press.

(>10 bars) and high-temperature (>200°C) molding processes including SMC and BMC.

It is difficult to provide any reliable estimate of tooling cost without detailed information of the size, complexity, and molding conditions. However, the cost of a simple one-sided composite tool is in the region of \$5000–\$10,000 for a tool of 1-m^2 plan area. Matched tools would cost $2\frac{1}{2}$–3 times as much, and metallic tooling 5–10 times. These are substantial costs and contribute significantly to the cost of the product.

2.5 Rate of Build

The rate at which a part can be laid up is a vital aspect of productivity for all laminated composite structures. This is determined by the method of lay-up and



1. Laminate on male tool

2. Press closed - compressed elastomer consolidates laminate during cure

Fig. 11 Pressure consolidation may be effected by use of single-sided tooling with an elastomeric counter tool setup in a press. For parts of moderate curvature and draw a simple block of elastomer may suffice. More deeply formed shapes will require the counter tool to be shaped. The elastomer tool is normally a much less costly option than conventional matched tooling.

the areal density of the reinforcements. Most construction is still hand laid and therefore the choice of reinforcement is the most important factor. The labor costs of laminating tend to be one of the critical cost factors, and this is strongly influenced by the number of plies that need to be laid. To maximize the build rate and to minimize lay-up costs, a heavy reinforcement is preferable, but infiltration, consolidation, and drape may be compromised if the reinforcement is too heavy. In the case of thinner laminates the specified configuration may require several plies to be laid at different orientations. Thus, if a quasi-isotropic laminate is specified, there must be a minimum of 8 plies of uniaxial reinforcement (e.g., prepreg) in the configuration $[0°, 90°, \pm 45°]_s$ or four plies of balanced woven fabric in the sequence [0°/90°, ±45°, ±45°, 0°/90°]. This leads to a minimum thickness of 1 mm if standard uniaxial or woven prepreg is used. A heavier reinforcement such as a woven glass fabric of 1250 g/m^2 would give a molded thickness of 1 mm for one layer, but the only configurations would be 0°/90° or ±45°. Alternatively two plies of a four-layer [0°, 90°, ±45°] noncrimp fabric of 625 g/m^2 would produce a 1-mm quasi-isotropic laminate. If drape were a problem, then a lighter-weight satin weave fabric, e.g., eight plies of 160 g/m^2, might be the better choice. For thicker laminates, where drape requirements are less severe, heavier reinforcements may be considered.

In the aerospace industry it is common to design complex laminates incorporating several thickness changes, cutouts, and other features. These must all be implemented while maintaining the basic configuration, balance, and symmetry of the laminate. This leads to the choice of thin uniaxial or woven prepreg, even for laminates that are very thick in their thickest regions. Laminates of over 20-mm thickness containing 160 plies are typically specified. This imposes a very considerable cost penalty in comparison with a simpler laminate made up from heavier reinforcements. The enhanced performance must be balanced against this cost penalty. There is currently considerable effort within the aerospace industry to develop manufacturing technologies that reduce processing costs, especially labor, while maintaining acceptable levels of performance.

An alternative approach is to use automated lay-up. This generally implies use of computer-controlled tow or tape laying equipment. For laying tape the machinery consists of a moving gantry with a tape laying head with 4–6 axis positional control. This is controlled by software linked to a computer-aided design and manufacturing (CAD/CAM) package to lay down a series of strips of tape to comply with the specified lay-up and part geometry. This equipment is suitable for making flat or shallow curvature panels, which may be sometimes subsequently further shaped. The equipment is costly but once set up, completely automatic. Quality and reproducibility are very good, but production rate is not always much higher than for hand lamination. This is due to the use of quite narrow tape, 50–600 mm, and a laying speed of only 1–5 m/s. The economics are generally more advantageous for the manufacture of large panels, e.g., >2 m length, where manual positioning of large sheets of reinforcement is difficult.

2.6 Economic Implications of Choice of Feedstock

A wide range of feedstock options are available; these range from the raw fiber tows in spool form, through the range of woven and nonwoven sheet materials, to the precompounded materials such as prepreg and SMC. There is an added cost associated with every operation performed on the fiber to convert it into

Table 3 Typical Costs of Basic Fiber Rovings (2000)

Fibre Type	Typical Weight (tex)	Price Range (US$/kg)	Approximate Annual World Production (ton)
E-glass roving	1,200	2–5	4 million
Aramid roving (yarn) (e.g., Kevlar 49)	300	10–20	10,000
High-strength carbon (e.g., T-300)	900	5–20	18,000
Intermediate modulus carbon (e.g., T-800)	350	100–200	<2,000 (estimated)

the feedstock of choice, and this must be offset against possible savings in the downstream processing operations. If dry reinforcement is used, then the matrix resin must be separately acquired and downstream processes must involve infiltration of the reinforcement. The alternative would be to purchase preimpregnated feedstock, such as prepreg, at a cost premium and then eliminate the need for infiltration downstream. Similar arguments may be used for the use of multilayer noncrimp fabrics in place of simple cloth or mat. In this case the lay-up process would be simplified at the cost of using more expensive feedstock. Typical costs of some of the more widely used feedstock materials are summarized in Tables 3 and 4. These values should be used with caution as actual prices vary widely according to availability, demand, the exact specification of the material, and, of course, the quantity to be purchased. The likely availability is indicated by the annual production figures. Thus E-glass is a commodity material and is readily available in all formats, but intermediate modulus carbon must often be specially ordered. The values quoted in Table 4 are based on actual quotations, obtained in 1998 and adjusted for inflation. Close analysis reveals several anomalies, but these are due to market forces at the time of quotation.

3 CONTACT MOLDING

3.1 Principles

Contact molding is the original process used for the manufacture of composite laminates; it is still very widely used and there are many modifications and

Table 4 Typical Costs of Converted Fiber Reinforcements—US$/kg (2000)

Reinforcement Format	E-glass	Aramid (Kevlar 49)	Carbon (T-300)	Carbon (T-800)
Fiber roving	3.75	15	15	120
Chopped strand mat	5.0	N/A[b]	N/A	N/A
Woven fabric	24	43	44	108
Noncrimp fabric	38	63	65	147
Uniaxial prepreg[a]	65	95	135	220
Sheet molding compound[a]	7.0	N/A	N/A	N/A

[a] These formats include the resin.

[b] N/A, not usually available in this format.

derivatives of the basic process. A single-sided tool is used, and this may be of either male or female form. The lamination surface of the mold is usually smooth and glossy, it is treated with suitable release agents to prevent adhesion of the laminate. These release agents are typically waxes and either silicone or poly(tetrafluoroethylene) (PTFE) suspensions. In the basic process, a layer of sheet reinforcement is laid on, or in, the mold, and then liquid resin is applied by brush or spray. The resin is worked by hand using brush or roller so that a uniform spread of resin and full impregnation, *wetting out,* of the reinforcement is achieved. A second layer of reinforcement may then be applied and resin worked into that layer as before, and so on, until the required number of plies have been laid. If a smooth glossy and/or colored surface is required, it is usual to brush or spray a *gel coat* consisting of a layer of a thixotropic resin with filler and/or pigment on to the prepared mold surface, and to allow it to gel before the first layer of reinforcement is applied. The most usual choice of resin is an unsaturated polyester (UPE). This is of a syrupy consistency and is formulated to cure at shop temperature, e.g., 15–25°C. When the resin is fully cured, the part may be removed from the mold, the mold prepared for another molding, and the process repeated.

3.2 Key Factors and Variations

Choice of Resin

Unsaturated polyester resins are very versatile, many variants are available, and their processing characteristics may be further modified at the point of molding by variation of the choice and proportions of initiator and accelerator added to the basic resin. The initial choice of resin involves its chemical formulation, viscosity, basic cure characteristics, and fire and smoke characteristics. The main resin types are ortho-phthallic, iso-phthallic, tere-phthallic, and bisphenol-based resins. The ortho-phthallic resins are economic general-purpose materials; iso-phthallic and tere-phthallic are premium grades offering superior performance especially in wet environments. Bisphenol resins are used for superior resistance to a number of chemical environments. Detailed recommendations are available from the suppliers. Most resins can be supplied with fire-retardant additives to improve their behavior when exposed to fire hazard. Fillers and pigments may also be incorporated in the resin to provide self-coloring and also to improve the surface smoothness, especially if the part is to be subsequently painted. It is generally considered that solid fillers and pigments impair the resistance of the resin to environmental hazards, so that unfilled and unpigmented systems are preferred when the component is destined to be exposed to water or chemical hazards, e.g., the underwater areas of yachts and motor boats. Viscosity is an important factor. Low viscosity improves the rate of wetting out and infiltration but can lead to resin *run-off* from vertical molding surfaces. A consistency similar to that of a medium lubricating oil is a common compromise, and there are often additions to promote thixotropy; this property allows the resin to thin when worked, e.g., with a brush or roller, but to thicken again when the working operation ceases. This allows good wetting out and reduces run-off. Alternative resin systems include the vinyl-ester and epoxy resins. The former may be processed in essentially the same way as the UPE resin and offer superior properties under some service conditions. The epoxy resins are considerably more costly than UPE, they are superior for higher temperature operation, and while widely used, they are not often processed by contact molding.

Resin Formulation

The resins are formulated by blending an initiator and often an accelerator with the basic resin. The initiator (often designated catalyst in the industry although most are not strictly chemical catalysts) is always added just before processing. The accelerator is often added to the resin by the supplier but may also be added at the point of processing. *Accelerators and initiators must never be mixed directly owing to the risk of a violent chemical reaction or explosion.* The accelerator is first thoroughly mixed into the resin, and then the initiator may be added and also thoroughly mixed. The combination of resin, accelerator, and initiator set the cure temperature range, the cure rate, the pot or shelf life, the gel time, and the cure time. Unsaturated polyester resins can be formulated to cure in times varying from less than 1 minute to over 24 h. The formulation must ensure that the resin remains workable, i.e., liquid and not too high a viscosity, for as long as it takes to lay and wet-out at least one layer of the reinforcement. In the case of small moldings, which might be completely laid up in an hour or two, it is common to formulate the resin to remain workable through the whole laminating process, but for large moldings the laminating operation may extend over several days and the resin is allowed to gel after each layer has been applied. However, it is imperative that not too long a period elapse before further layers are applied, otherwise bonding may be inadequate. Typically, a period of up to a week is permitted. At the end of the resin working period gelation occurs quite suddenly. The resin ceases to be liquid and, although still quite soft for a period, any attempt to work it would result in resin fracture. Once the laminate is complete, it is generally desirable that the resin cures in as short a time as possible. The molding may then be removed from the tool, *demolded,* and the tool may then be reused. Typically if gelation takes place after 1 h, then demolding will be possible after about 5 h.

Cure Temperature and Cure Time

Unsaturated polyester resins with no added accelerator need to be cured at elevated temperatures in the range of 70–160°C. The exact temperature of cure in this range is determined by the choice of initiator, several of which will be recommended by the resin supplier. They have storage lives of upward of 1 year in a dark cool environment. The addition of an accelerator is required for cure at shop temperature, when satisfactory cure may be affected at temperatures above 15°C, although 20–25°C is usually considered more satisfactory. Increasing the proportion of initiator results in a faster cure. UPE resins can be cured in less than 1 min, even at 20°C, or the cure extended to several hours. An alternative strategy is to formulate the resin to cure at an elevated temperature, e.g., 50–120°C. This will ensure a very long working life at shop temperature. After the lay-up is completed, the molding must be heated to its cure temperature, and cure can be much faster than would be achieved with a low-temperature cure. Heating may be by placing the tool in an air-circulation oven, by radiant heaters, or by blown hot air. The need to ensure adequate working life has already been discussed; the other critical consideration is exotherm (see also discussion of cure of thermosetting resins above). The amount of heat released during cure is approximately the same for most UPE resins, so the rate of heat evolution will be a function of cure rate. Faster cure will result in a higher rise in temperature. This will also be affected by the thickness of the molding and by heat transfer from the laminate to the mold and the environment. Fast cures can often be tolerated in thin laminates but in thick sections would result in unacceptable temperatures being reached. It should be noted that exothermic heating occurs even in *cold* (i.e., shop temperature) cured systems. The temperature of a molding will typically be observed to rise to 50–80°C. The cure is actually accelerated by this temperature rise, and thin laminates will take longer to cure than thicker ones where the exotherm effect is more severe. In practice the proportions of accelerator and initiator should be adjusted for each type of molding to optimize working time, curing time, and hence productivity.

Styrene Emissions

Unsaturated polyester resins are formulated with a proportion of styrene or other vinyl monomer that is used to control the resin viscosity and provide the basis for the addition cure. Styrene is highly volatile, and inflammable, and the vapor is known to constitute a health hazard. Hence the permitted levels of styrene in the working environment are strictly regulated by the health authorities. Details vary according to location, but there will always be a requirement to provide sufficient ventilation and regulate the release of styrene vapor into the atmosphere. Lamination of UPE resins onto open molds obviously constitutes an extreme hazard. Resins are available with film-forming additives that help limit emissions, but there is a movement to adopt alternative processes that reduce potential exposure.

3.3 Laminate Design for Contact Molding

The contact molding process is extremely versatile. The size of moldings varies from less than 1 kg up to more than 500 tons, and many features can be incorporated into the molding. It is suitable for production runs from single parts up to several hundred but is basically a low production rate process and is dependent on a high labor content. Quality is also strongly dependent on the expertise of the labor employed.

The cheapest reinforcement is chopped strand E-glass mat. It is of low V_f but high permeability and has good drape characteristics. It is therefore very widely used for production of commodity moldings. Its disadvantage is that the mechanical properties are only modest, and it is therefore not ideal where structural mass is an important consideration. It is available in a range of areal densities, generally from about 100 to 5000 g/m^2 and may be treated with different proportions and types of binder. Resin-soluble binders allow the mat to wet-out and drape very easily once the resin is applied, while insoluble binders stabilize the mat through the wet-out process. The choice of weight and binder depends on the complexity of the molding. Superior mechanical properties are obtained if woven or noncrimp fabrics are selected or by the use of aramid or carbon reinforcements. These are, of course, more costly and also less permeable. This renders them more difficult to wet-out and extends the lamination time, reducing productivity. An excellent general compromise is to alternate layers of CSM or CRM with woven or noncrimp fabric. The random mats help distribute the resin and speed up the laminating process while the woven materials enhance the mechanical properties. Some typical lamination strategies are illustrated in Figs. 12–14. Mechanical properties, especially bending and/or torsional stiffness can also be enhanced by designed-in features such as ribs, hollow or cored stiffeners, and cored sandwich sections. Metal or timber inserts for local stiffening or for subsequent attachments may also be easily incorporated. Some of these possibilities are illustrated in Fig. 15.

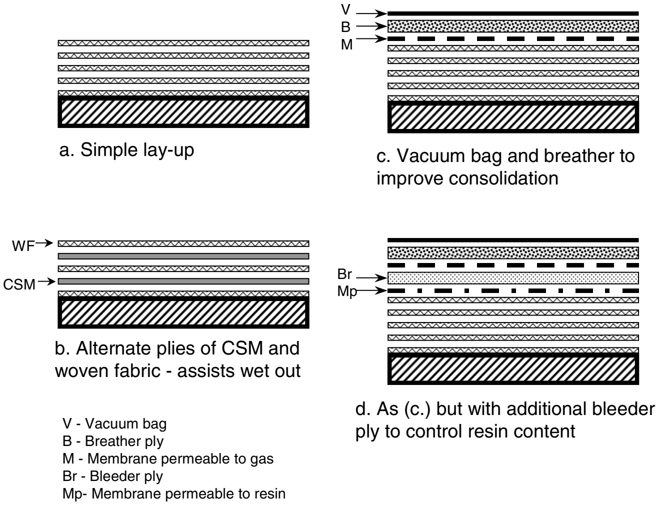
3.4 Some Variations on Basic Contact Molding Process

Vacuum Bagging

When the lamination process is complete the laminate is covered with a flexible bagging film, usually a polyamide or elastomer. This is sealed around its periphery to the mold and the space between tool and bag is connected to a vacuum system. This applies a consolidating pressure of up to 1 atm (≈1 bar) to the laminate, assisting consolidation, improving surface finish on the *back* surface, and reducing styrene emissions. It is advantageous to place a porous breather blanket between the surface of the molding and the vacuum bag (Fig. 12*c*) to ensure that the vacuum is evenly distributed over the surface and that any wrinkles forming in the bag material are not impressed on the surface of the part.

Prewetting Out Reinforcement

When manufacturing large moldings, it is often convenient to prewet the reinforcement before laying on the mold. This may be accomplished by passing a



a. Simple lay-up

c. Vacuum bag and breather to improve consolidation

b. Alternate plies of CSM and woven fabric - assists wet out

d. As (c.) but with additional bleeder ply to control resin content

V - Vacuum bag
B - Breather ply
M - Membrane permeable to gas
Br - Bleeder ply
Mp- Membrane permeable to resin

Fig. 12 Alternative strategies for lay-up of contact molded laminates. (*a*) Simple lay-up of similar plies, mat or fabric. (*b*) Plies of chopped strand mat (CSM) are alternated with woven fabric. The woven fabric has a much higher fiber density and continuous fibers and thus gives higher stiffness and strength than mat. The mat is more permeable and assists wet out of the fabric. (*c*) Breather and vacuum bag are applied to provide better consolidation. (*d*) Further bleeder layer has been incorporated to absorb excess resin. This helps control the resin content.

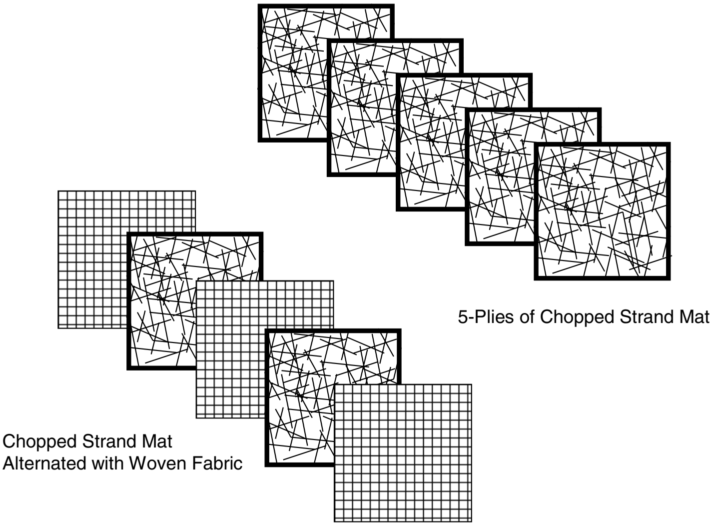


Fig. 13 Further illustration of the use of alternate plies of mat and fabric.

roll of reinforcement, typically 1–2 m wide, through a trough of liquid resin and then passing it through one or more pairs of rolls, which ensure uniform impregnation and control the final resin contact. The wetted reinforcement is then laid into or onto the mold and cut to length as required. This speeds up the process and is more convenient than handling large sheets of reinforcement. The hull moldings of larger vessels are laid this way. Rolls of woven roving are

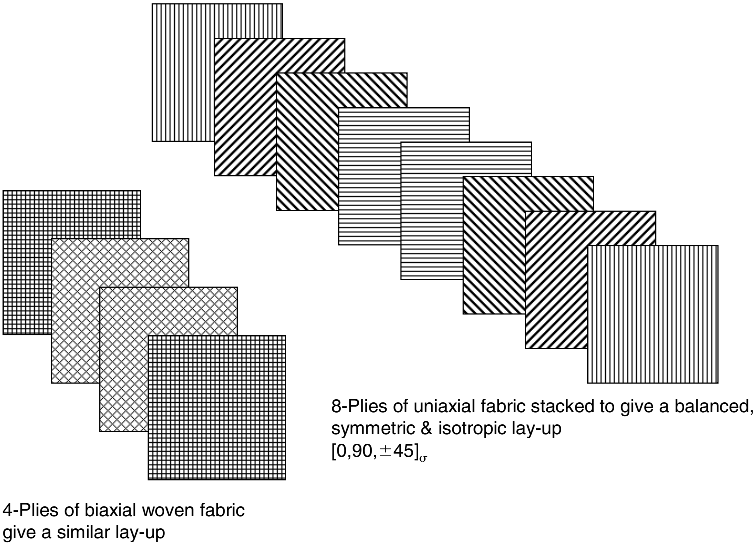
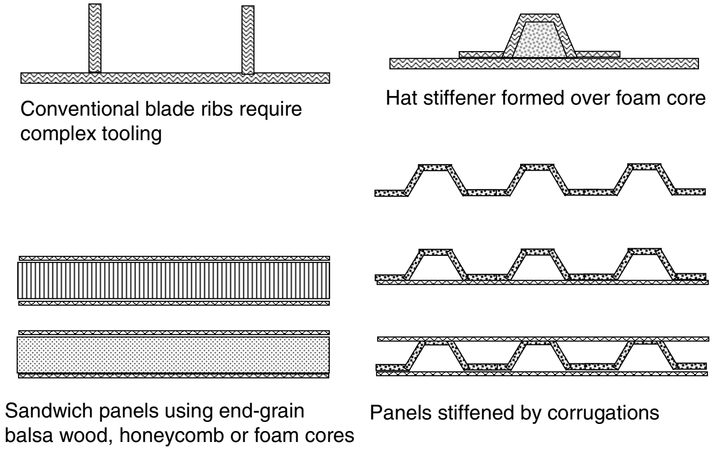


Fig. 14 Uniaxial reinforcement, such as prepreg, must normally be laid up in a balanced, and symmetric sequence. This requires eight plies to give an isotropic laminate. If balanced woven reinforcement is used a similar effect may be realized with just four plies.



Conventional blade ribs require complex tooling

Hat stiffener formed over foam core

Sandwich panels using end-grain balsa wood, honeycomb or foam cores

Panels stiffened by corrugations

Fig. 15 Laminates may be effectively stiffened in a number of ways. Conventional blade stiffeners are effective but require more complex tooling. A simple method is to laminate extra material over foam or timber former to form a hat section. Sandwich sections are very efficient and can be formed using end-grain balsa wood, aluminum, or aramid honeycomb and foam cores. Corrugations may be incorporated into the tooling to provide stiffening and more complex double and triple layers are a further possibility.

laid from gunwale down to the keel and back up to the opposite gunwale. The layers are consolidated by hand or mechanical rolling. The largest hulls manufactured this way are some 85 m long and weigh upward of 250 tons.

Spray-up

The contact molding operation can be further mechanized by use of the spray-up process. The basis of this is a spray head from which liquid resin and chopped rovings are sprayed simultaneously onto the mold surface. Continuous fiber rovings are fed into the spray head, which is equipped with a cutting device that cuts them to lengths of 25–75 mm; a jet of compressed air directs the chopped fragments onto the mold. Resin and initiator or curing agent are also fed separately into the spray head, where they are blended in the correct proportions and sprayed so that they wet out the chopped rovings on the mold surface. The resin/fiber layer on the mold must then be consolidated by rolling. The spray head may be directed by hand or further automated by use of a robotic manipulator. It is convenient to program such robots by having them replicate the actions of an expert sprayer. This is a cheap, flexible, and effective programming technique. The principal disadvantages of the system are that only random layers can be deposited and that styrene emissions are very high. To combat the emission problem, hand spraying must be conducted in adequately ventilated booths, but robotic spraying could be carried out in completely sealed environments. The random layer limitation may be partially overcome by interrupting the spray process and laying intermediate layers of woven fabric and applying the resin by spray, followed by a further layer of the chopped material.

3.5 Productivity and Economics

The contact molding process is attractive because it is very versatile, cheap tooling can be used, and production runs as low as that of a single molding may be viable. Quality is dependent on the skill of the labor force and the process is slow. It is eminently suitable for the production of very large moldings, where long runs are unlikely. For smaller moldings there are few circumstances where more than one part may be manufactured from a single tool set in a working day. Ordinary composite tooling, under favorable manufacturing circumstances, may be sufficiently durable to produce 100 parts before the tool requires extensive rework or replacement. A run of 1000 parts per year would thus require about five tool sets in constant operation and would consume at least 10 tools per annum. A labor force sufficient to handle this volume of work would also be required. This means that where larger production runs are envisaged alternative processes that are more reliable, in terms of quality, and less dependent on high labor content must be sought.

4 PRESS MOLDING

4.1 Basic Process

The principle of press molding is that a pair of matched tools and a press are used to apply pressure during consolidation and cure. Within this scenario there are several variants depending on the size of the molding, the pressure used, and the cure temperature. The advantage of matched tooling is that the mold cavity is more precisely defined, section thickness is more accurately controlled, and a high surface finish may be imparted on all surfaces of the molding. The pressures used to consolidate the part vary considerably according to the projected area of the mold, and the tooling must be designed to be sufficiently robust. If low pressures, <5 bars, are used, then composite tooling with solid support structure, e.g., cast-filled resin or concrete, may be adequate. At higher pressures metal tooling is needed. Aluminum or zinc may be adequate for moderate temperatures and cure temperatures, but steel will be required at the higher end of the pressure/temperature spectrum. It should be noted that a more precise control of dimensions and surface finish, as well as more durable tooling is achieved if steel tooling is used. Molding surfaces may be further hard coated, e.g., hard chromium plated, to enhance surface finish and durability.

4.2 Reinforcements and Resins for Press Molding

All conventional dry and preimpregnated reinforcements may be used in press molding. However, it is highly desirable to minimize lay-up time in the press because this would restrict productivity while the equipment remained idle. For this reason it is common to preform the reinforcement so that a single entity may be placed in the mold. The simplest preforms are made by spraying chopped fibers with a small proportion of binder onto a perforated former on a vacuum box. The loose preform may be briefly consolidated by vacuum bag or by an auxiliary pressing operation at low pressure and moderate temperature, usually <100°C. Fabrics may be tailored to size and preconsolidated in a similar way. Again a small proportion of binder is necessary, and this may be incorporated in the fabric at the point of manufacture or applied as a spray or dry powder. In either case the preform is simply placed in the open mold, and the molding operation is then initiated.

Either ambient temperature or elevated temperature cure resins may be used. The characteristics required of the resin depend on the chosen method of infiltration. The resin may be preimpregnated into the reinforcement either before, during, or after preform manufacture. Alternatively the resin may be placed in the mold with the preform as a liquid or sometimes as a film or sheet. A further possibility is that the resin is injected into the closed mold; this is resin transfer molding (RTM) and is discussed in a later section. Any of the common thermosetting resins may be used; UPE and vinyl-ester systems are the most common. To minimize the molding time, a fast curing system is desirable, subject to full infiltration and control of the exotherm during cure.

Press molding is widely used with precompounded reinforcements such as prepreg, sheet and bulk molding compounds (SMC, BMC), and also with thermoplastic matrix systems, e.g., glass-mat/thermoplastic (GMT), these are discussed in later sections.

4.3 Ambient Cure Press Molding

In this version of the process low pressures and unheated tooling is used with a very fast curing resin formulation. A typical sequence of operations is that the preform is loaded into the open mold, a carefully metered quantity of the resin, which must be of low viscosity and have been just mixed with the initiator, is poured or sprayed into the mold. The mold is then closed and the consolidating pressure applied. This forces the liquid resin to completely infiltrate the preform. Vents are often provided to allow a little excess resin to escape, but the process depends on the hydrostatic pressure generated in the resin to effect the infiltration. The mold remains closed and under pressure until the resin is sufficiently cured. This may take anywhere from 2 to 20 min depending of size and complexity of the molding. The fast curing resin generates a considerable exotherm, so that the molding is heated, often to more than 100°C, during the cure process. When the cure is judged to be adequate, the press is opened and the (hot) part is removed. The next preform may then be inserted and the process repeated.

The attraction of this process is that the capital investment of press and tooling is relatively low and the production rate quite fast. Simple composite tooling is adequate for short runs. Surface finish may be enhanced by using a quick gelling sprayed gel coat. The process is not generally suitable for large moldings, e.g., $>2\ \text{m}^2$ projected area, and control of dimensions and porosity is relatively poor. It is very suitable for moderate runs of commodity items where high mechanical performance is not a requirement.

4.4 Elevated Temperature Cure Press Molding

In this case metal tooling incorporating internal heaters must be used and, generally, more substantial presses and higher consolidating pressures are used. The cure temperatures are typically 80–160°C for UPE and vinyl ester (VE) systems and up to 250°C for epoxy and some high-temperature resins. The general procedure is similar to that described above for ambient cure. However, there is much more flexibility as a fast curing (at ambient temperature) resin is not necessary. Resins are formulated to cure in 2–20 min at the relevant cure temperatures. They then have reasonably long pot lives at shop temperature. Preforms are again preferred and the resin applied as liquid or film. The press is closed, and, as the resin is heated, it first becomes less viscous so that infiltration is accelerated. The application of pressure is often programmed during this period, typically a few minutes. When the charge has been heated and the full pressure applied, the resin is allowed to cure. Again, consideration must be given to dissipation of the heat generated during cure, but generally conduction to the metal tooling is quite effective in preventing hot spots and *burning*. Surface finish may, again, be enhanced by use of a gel coat, and a further option is to slightly open the mold after gelation of the main charge and to inject a further quantity of pigmented resin between the workpiece and the mold wall, sufficient to form a surface layer 0.5–1.0 mm thick. The pressure is then reapplied while this injected gel coat is cured. At the completion of the cure, the press is opened and the hot part is ejected. Average times for the complete cycle are 10–20 min. This is much faster than can be achieved with contact molding, and there is the additional benefit of closer tolerances, better surface finish, and better quality control. The negative aspects are higher investment in tooling and equipment and size limited to the sizes of the available presses, e.g., 2 × 1 m plan form.

5 AUTOCLAVE PROCESSING OF PREPREG

5.1 Autoclave

An autoclave, shown schematically in Fig. 16, is a pressure vessel capable of being internally pressurized with a gas, which can also be independently heated. This is in contrast to medical autoclaves, which are usually steam operated. Modern industrial autoclaves are often very large, commonly up to 3 m internal diameter and 4–6 m in length. They are pressurized with nitrogen gas typically to pressures of up to 5 bars. The use of nitrogen, rather than air, avoids any chance of combustion of hot resin or other flammable materials. The contained gas is mechanically circulated and may be heated to temperatures of up to 400°C, although ≈200°C is a more common upper limit. There are sometimes also

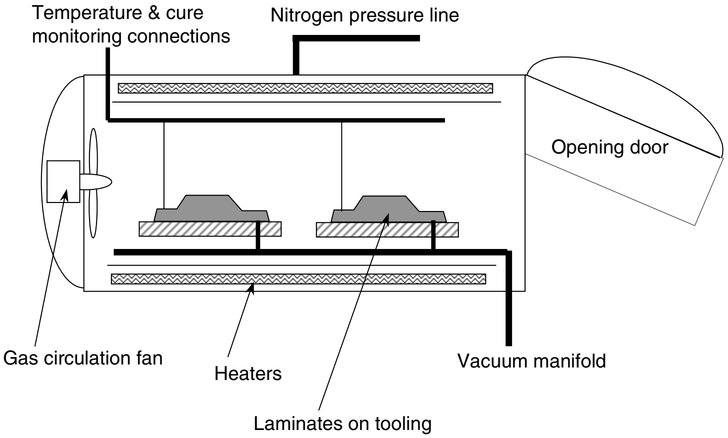


Fig. 16 Industrial autoclaves vary in size from less than 1 m diameter to more than 4 m, and in some cases more than 10 m in length. They are substantial pressure vessels. The interior is pressurized with nitrogen and may be heated, typically up to 250°C. A vacuum manifold and monitoring connections are provided to attach to the individual tools.

means for cooling the interior. The autoclave with its associated pressurization, heating and control equipment constitutes a considerable capital investment, typically in excess of $1 million. This has a significant influence on the cost of autoclave processed parts. It also occupies a lot of factory space. For these reasons autoclave processing is mainly used for the manufacture of premium products in high-performance composites.

5.2 Prepreg

Prepreg is the common name for *preimpregnated warp sheet:* It was briefly described in Section 1.2. It consists of a uniaxial array of fiber tows or roving, formed by butting up a large number of individual tow ends (i.e., fiber from many spools). The web thus formed may be from 300 mm (*tape*) to 2000 mm or more, (*broadgoods*) wide. It is supported on a carrier film, usually silicone-treated kraft paper, which has been precoated with a layer of resin. A second layer of film, also coated in resin, is applied to form a sandwich, which is passed between a number of heated rolls and then, often, through chilled rolls. This thoroughly impregnates the fiber web and produces a uniform material. The objective is to incorporate exactly the correct proportion of resin into the prepreg, e.g., to form a composite with $V_f = 0.6$. Sometimes a small excess of resin is incorporated so that some may be bled off during cure, but modern practice is not to bleed (see subsequent discussion) but to use so-called *net resin* materials. Standard prepreg is manufactured to be 0.125 mm ($\approx$10 mils) thick after processing. Alternative thinner prepreg is sometimes available, mainly for space applications and a woven form with a thickness of 0.25 mm per ply is becoming very popular. Prepregs are manufactured from several grades of carbon fiber, aramid, E-glass, S2-glass, silica (“quartz”), and some ceramic fibers. Carbon and aramid are the most widely used. The resin used in prepreg is formulated to be pasty at shop temperature with a controlled degree of *tack* (stickiness). This allows the sheets to be accurately tailored and then placed, by hand or machine. Too much tack renders it very difficult to handle (it sticks to everything!). Too little tack makes it more difficult to locate on the tool. The prepreg is cured by heating, and pressure is used to effect consolidation (see Section 2.2). The prepreg is supplied in continuous rolls, typically 600–1000 mm wide and up to 100 m long. It is usually necessary that the prepreg be stored in a cold room (< -15°C) to maximize storage life.

5.3 Tooling

The most common form of tooling is to use single-sided molds with vacuum bag consolidation. The tools are usually made from composites, with low-temperature carbon fiber/epoxy being very popular. These tools are relatively cheap, robust, and have thermal expansion characteristics that match that of carbon fiber composites. Since the process is very widely used in the aerospace industry, where one side of the molding will be the aerodynamic surface, it is common practice to define this shape at the mold surface. Features such as stiffeners and ribs are then built up on the inside surface of the molding using auxiliary mold fixtures. These can become very complex but allow for the manufacture of large intricate moldings, which confer the benefits of *parts consolidation.*

5.4 Cutting the Prepreg

Prepreg may be hand or machine cut. The latter is adopted for all but the smallest operations. The prepreg rolls must first be withdrawn from cold storage and allowed to equilibrate to shop temperature. This may take up to 24 h. The roll must not be removed from its protective bag or unrolled while it is cold, otherwise moisture may condense from the atmosphere onto the cold prepreg. This would lead to water absorption and processing problems, e.g., porosity. Cutting should be carried out in a clean room with temperature and humidity controlled [20°C and 50% relative humidity (RH) is common]. For hand cutting shears, a *Stanley* knife, or a cutting wheel may be used. It is usual to make templates for each ply from card, plastic, or sheet metal to provide a cutting guide. Each template must also indicate the fiber orientation (e.g., 0°, 90°, ±45°) so that the template may be correctly positioned on the roll of prepreg. When the width of the part exceeds the width of the prepreg, it may be necessary to cut several pieces to make up a single ply. Many moldings are of varying thickness. This involves ply drops, which are preferably located toward the center thickness so that there are no steps on the outside surfaces. It will be apparent that the logistics of laying up a complex laminate of, say, 48 plies, with a specified configuration, (e.g., the quasi-isotropic [0°, 90°, ±45°]s), will be quite complex. Each ply will require a drawing, part number, and template. The plies must then be cut from the roll of prepreg, with due regard to minimizing wastage; each ply must be identified and then stacked to form a kit of plies in the correct order for the lay-up operation. If necessary positional index marks must be placed on the cover film of each ply to facilitate precise positioning. These are the arguments for using an automated cutting system.

The automatic prepreg cutter consists of a flat bed, typically 2 m wide by 10 m long. The prepreg sheets are unrolled along the bed, butting two or more together if a wider strip is required and, sometimes, more than one layer. They are retained in place by suction from under the bed. The cutter head is mounted on a gantry that moves along the bed in the length direction and may also traverse in the width and vertical directions. The cutting method may be a reciprocating knife, ultrasonic knife, or water jet. The cutting operation is controlled by a computer that is linked to the CAD system. Thus, each ply is defined in the software, so neither drawings or templates are required. The software also incorporates a nesting program that adjusts the cutting sequence to minimize waste. The software and the machines have been developed from those used in the garment industry. A small automatic prepreg cutter is shown in Fig. 17. The cutting operation is initiated and the cutter automatically cuts all the plies defined on the bed. There is also a printing head that numbers each piece and can print index marks that are used to facilitate precise lay-up. At the end of the run the cut plies are gathered and collated, either by hand or by a robotic device that automatically collects and collates the plies into kits ready for lay-up. Although the cost of such equipment is high, the benefits in terms of precise cutting, speed, elimination of hand operations, waste minimization, and improved quality assurance are very considerable.

5.5 Hand Lay-up

The tool is prepared and a release agent and/or release film applied across the molding surfaces. The prepreg kit is delivered to the lay-up location. At this

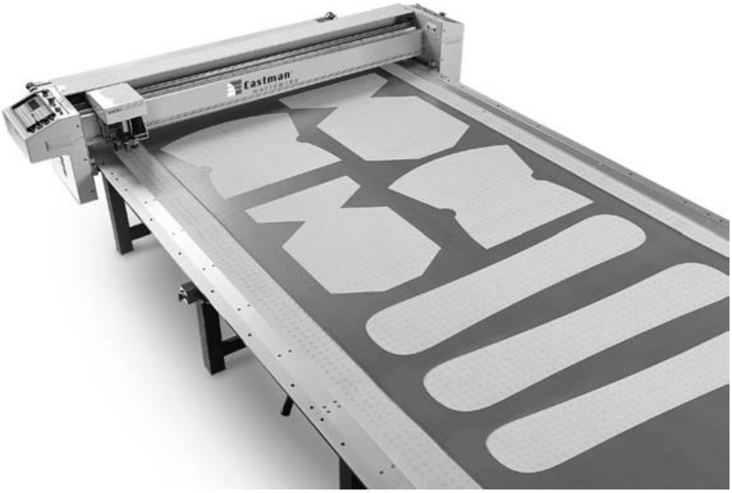


Fig. 17 View of a small automatic prepreg cutting machine. This shows how the plies have been positioned to minimize wastage. (Photograph by courtesy of the Eastman Machine Co., Rochester, NY, Model M9000)

stage each piece of prepreg is still protected by the plastic or paper film on each side. The pieces are taken in the correct sequence, the protective film removed from the mold-face side, and the piece carefully positioned on the tool. Once its correct location is confirmed, It may then be partly consolidated by use of hand, brush, or roller. Special care is taken to avoid any wrinkling or pockets of entrapped air. (The tack allows the ply to be repositioned, if necessary, before consolidation but not once it has been pressed down.) The outer protective film may then be removed and the next ply positioned. A typical lay-up sequence is depicted in Fig. 18. It is usually necessary to further consolidate, or *debulk,* after

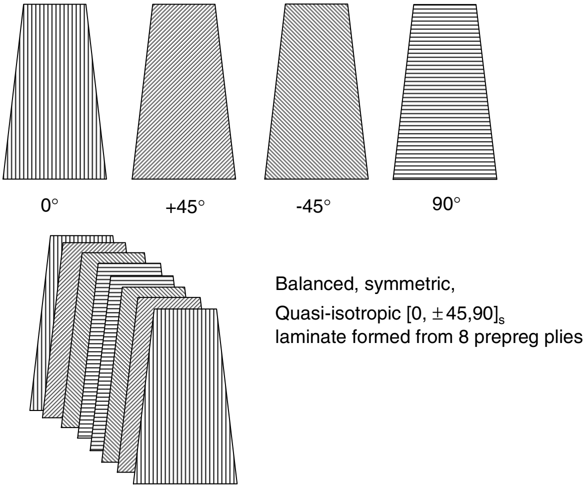


Fig. 18 Illustration of the necessary prepreg ply shapes showing fiber orientations for laying up a quasi-isotropic laminate.

every 12, or so, plies have been laid. This is done by placing a vacuum bag over the mold, evacuating and usually warming to 50–70°C for a period of the order of 60 min, using an oven or radiant heaters. This softens the resin and allows the pack to consolidate under the pressure exerted across the bag. The next set of plies may then be placed until the lay-up is complete.

In the case of smaller moldings the prepreg pieces may be handled quite easily and positioned by eye. If larger pieces, >2 m, are to be placed, some form of mechanical assistance may be required. Manipulators with suction pads, which operate on the back cover film, are convenient. For precision placement index marks must be printed on the back-cover film and a computer-controlled laser projector, set above the lay-up location, used to project reference spots, a red spot, or cross onto the back of the laminate. Each ply is lined up with these reference spots and may be precisely located. The back-cover film is then removed and the computer set for the next ply.

5.6 Automated Lay-up

This is more difficult to accomplish than automated cutting and collating. In fact no fast, reliable method has yet been developed for laying precut plies onto the mold. The only system is the automatic tape laying machine. This may be based on a flat bed, e.g., 10 m × 2 m, with moving gantry and a tape laying head with 3–5 independent axes of motion. Prepreg tape, 50–600 mm wide, is dispensed into the moving head where it is unrolled, the end trimmed, the backing foil removed, and the tape laid onto the mold surface and rolled down to partially consolidate. The most sophisticated tape laying machines can lay the tape onto a complex tool or mandrel that is independently manipulated. This provides a great deal of flexibility in the shapes and lay-ups that can be handled. A photograph of one of these machines laying an aircraft fuselage component is shown in Fig. 19. Instead of complete plies the layers are formed by rows of tape, oriented according to the requirements of the specified configuration. There is minimal waste, just the portion lost when the tape ends are trimmed to fit the

Fig. 19 Large tape laying machine. Both the tape laying head and the tooling are manipulated to form a large aircraft fuselage component. (Photograph of the Viper tape layer courtesy of Cincinnati Machine Co., OH)

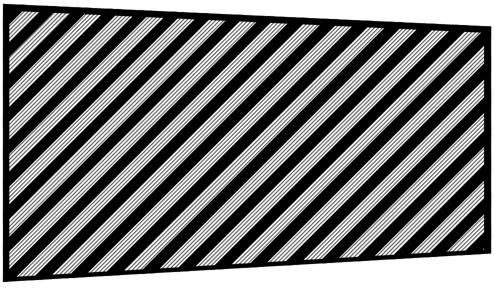
ply perimeter. Tape laying allows conformation to surfaces with a higher degree of double curvature than sheet reinforcements because small displacements may be accommodated between the strips of tape, allowing small gaps or slight overlap. A representation of a tape-laid ply is shown in Fig. 20. An alternative strategy is to use a separate machine to prepare the tape. This cuts the tape to the required lengths, trims the ends to the correct angles, and discards the waste. The prepared lengths of tape are transferred to a dispensing cartridge that is loaded into the tape laying machine. This eliminates the need for the laying head to trim the tape and is therefore faster. It also eliminates problems due to faulty end trim and the possibility of the small waste offcuts being transferred to the molding. The tape preparation equipment does not need the complex motions of the laying head and is, thus, cheaper and faster. While tape laying machines are automatic and require little manual intervention, they are not fast. Laying speeds are only about 1 m/s, not allowing for turnaround time at the end of each traverse. This is a consequence of the inertia due to the mass and size of the moving parts. Tape laying machines are large and again represent a considerable capital investment. Although initially troublesome, the equipment is now well developed and reliable and is considered to be more economic than hand lay-up for moderate runs of large moldings. Nevertheless automated cutting coupled with hand lay-up is still the most common practice.

5.7 Cure Monitoring Sensors

Temperature and cure monitoring sensors are commonly incorporated into all larger moldings or a proportion of smaller parts. These consist of fine wire thermocouples and/or proprietary cure sensors. These are smart chips that measure the changes in dielectric properties of the resin and can be used to assess the progress of the cure. They may be incorporated into parts of the molding that will be eventually discarded, but they are so small that they are unlikely to compromise the performance of the component.

5.8 Tool Preparation for Curing

When the lay-up process is complete and the molding has been sufficiently debulked, the mold must be finally prepared for cure in the autoclave. There are several variants adopted by different operators. The most common is: The pe-



45° ply formed by tape laying

Fig. 20 Single flat 45° ply as laid up by an automated tape laying machine. The ends of each strip of tape must be accurately trimmed to the profile of the ply.

riphery of the molding is sealed to prevent in-plane resin bleed. This is often done by fixing a self-adhesive cork dam to the tool face, around the edge of the part. A semipermeable release membrane is then placed over the part. This is most usually a fine woven glass fabric that has been coated in PTFE (Teflon). This will allow liquid resin and gases to pass through but will not stick to the cured part. It also imparts a fine woven texture to the back surface of the molding. A layer of porous material is then placed on top. This may be woven glass fabric, glass or polyester mat, or even blotting paper. It serves to absorb any excess resin bled from the curing laminate. The thickness of this *bleeder* layer is adjusted according to the quantity of resin to be bled. In many current operations, *zero bleed* or *net resin* systems are used, in these cases the bleeder may be omitted altogether and an impervious release membrane used. However, many consider it desirable to use some bleed. On top of the bleeder is placed a further perforated film, usually polyester or PTFE, which allows gas, but not resin, to pass. There is then a further layer of porous fabric, usually a polyester felt, termed the *breather*. This maintains a continuous gas-permeable layer over the molding. The final layer is the vacuum bag, a polyamide (nylon) or elastomeric film, which is sealed to the mold surface, outside the cork dam. This arrangement is depicted in Fig. 21. A vacuum connection is made, either through the bagging film or through the mold, so that a vacuum may be applied to the breather ply. When this is done, atmospheric pressure will act on the vacuum bag and press the pack onto the molding surface. The continuity within the breather and the permeable membranes allows gases and volatiles to be extracted from the entire pack under the bag. The vacuum is maintained while the mold is moved into the autoclave, when the autoclave vacuum system is attached.

5.9 Autoclave Operation

The autoclave is loaded with as many molds as it will accommodate. This might be just a single large piece, e.g., a major wing component, or several smaller

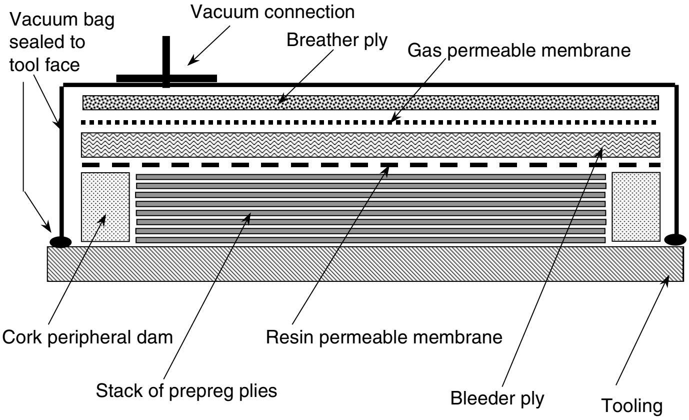


Fig. 21 Typical arrangement of prepreg laminate and auxiliary materials for autoclave molding. The perimeter of the laminate is sealed with a cork dam that prevents outward resin flow. A bleeder ply is set above a resin-permeable release membrane to absorb excess resin. The breather ply ensures a continuous open passage for the vacuum application. The whole assembly is covered with a vacuum bag sealed to the tool face. The vacuum connection is shown through the bag, but an alternative is to connect through the tooling.

panels. Each is connected to the vacuum system and temperature and cure sensors connected to the central monitoring point. The autoclave may then be closed and the cure cycle initiated. There are three controllable inputs to the cure cycle: vacuum, pressure, and temperature. A typical cycle is depicted in Fig. 22 and will now be described and discussed.

The autoclave is flushed out with nitrogen, to eliminate air, and may then be pressurized. The cycle commences with the vacuum line operating and the autoclave at ambient temperature and pressure. The heating cycle is started. As the resin is warmed, it softens allowing further consolidation to occur under the pressure induced by the vacuum. Eventually the resin becomes quite fluid and consolidation can be completed. At this stage the internal pressure is usually increased and the vacuum lines opened to atmospheric pressure. The consolidation is maintained by the difference between atmospheric pressure and the pressure inside the autoclave. The reason for this is that with the vacuum applied, the absolute pressure inside the molding is that of the vacuum. The autoclave pressure is carried by solid-to-solid contact between the reinforcement fibers, with the liquid resin effectively floating in the interfiber spaces. Any gas or vapor porosity, effectively a bubble in the resin, will tend to dilate under these conditions. By opening the space under the vacuum bag to atmospheric pressure, this porosity will contract. A further alternative is to pressurize both the autoclave and the space enclosed under the bag, always maintaining a higher pressure in the autoclave body, so as to maintain consolidation. This positive pressure reduces pores more effectively and can even eliminate porosity by forcing air and volatiles into solution in the liquid resin. It should be noted that the concept

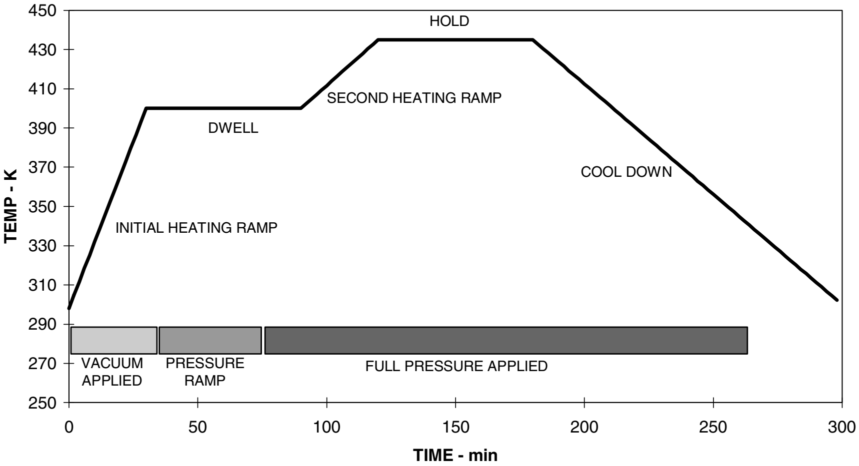


Fig. 22 Typical temperature profile for a prepreg cure cycle. The initial heat-up rate is controlled, typically 5–10 K/min. The vacuum under the bag is normally maintained during heat-up. A dwell is incorporated to assist temperature equalization in the laminate and to extend the process window. The vacuum system is typically vented to atmosphere at this time and the consolidating pressure applied in the autoclave. When consolidation is completed, the temperature is raised to the cure temperature. When cure is complete, the autoclave is allowed to cool down to ambient temperature, with the pressure still applied. Finally the pressure is reduced so that the autoclave may be opened and the parts removed.

of sucking away volatiles with the vacuum is flawed, as it would be virtually impossible for a bubble to pass through the tightly packed prepreg fibers and hence out through the bleeder and breather plies to the vacuum pump.

The point at which the autoclave pressure is applied can be critical. If the resin is very fluid, too much may be squeezed out of the laminate. However, in most systems the opposite problem dominates. This is to ensure complete consolidation and elimination of porosity before the resin gels. This can be exacerbated by the fact that the prepreg temperature will not be uniform. The outside is heated first while the interior remains cooler. Likewise the viscosity will be lower in the hotter regions, until the cure advances and the viscosity rises sharply to the gel point. The problem is to create a processing window where the resin is sufficiently fluid to allow consolidation to proceed to completion. This may be accomplished by introducing dwell intervals in the heating cycle, as shown in Fig. 22. The dwell or hold allows the temperatures to equilibrate in the molding and can be used to control the time at which gelation occurs. Another consideration is the cure exotherm, which can result in local overheating, even in quite thin laminates, e.g., 10 mm. This is because heat flow into and out of the laminate is very inefficient due to the insulating characteristics of the bleeder and breather plies in particular. A viscosity and cure profile is shown superimposed on the heating cycle in Fig. 23. Note that initially the resin viscosity drops as the laminate heats up. The dwell serves to extend the time that the resin remains at low viscosity, allowing consolidation and elimination of porosity to proceed. When the temperature is ramped up to the final cure temperature, the resin gels and viscosity increases sharply. At this stage the exotherm is most intense and local overheating can result. The heating rate must be controlled to prevent this overheating and, if necessary, a further temperature dwell is introduced. The processing window is the interval when the viscosity lies below an arbitrary level where effective consolidation will occur at the pressure employed.

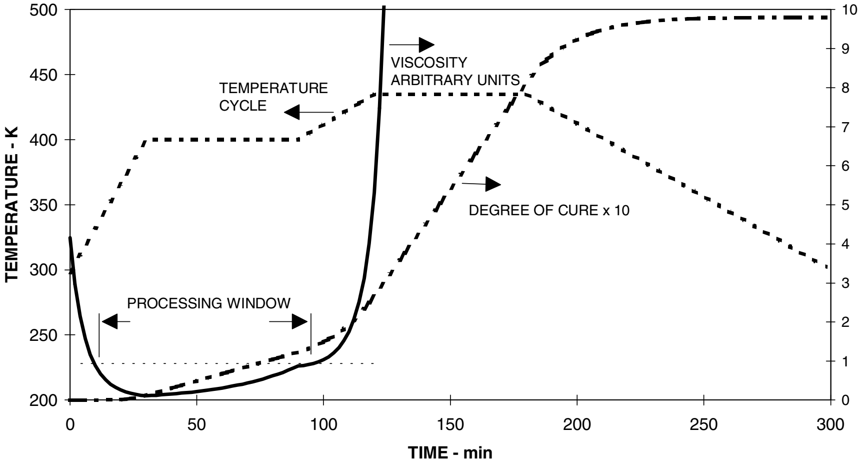


Fig. 23 Shows the cure and viscosity profiles for a typical epoxy system under the cure cycle described in Fig. 20. Note how the period of low viscosity is extended by incorporation of the initial dwell at 400 K.

Once the resin is sufficiently cured, the heaters are switched off and the autoclave cooled down to ambient with the pressure maintained. When sufficiently cool, the pressure is reduced, the interior of the autoclave flushed with air, the doors opened, and the molds removed. The whole process is likely to have extended over 12–24 h. It will be clear that it is no simple matter to set the cure cycle. It should also be appreciated that the cost of materials, labor, and other added-value items making up an autoclave load is very high, so that faulty treatment must be strenuously avoided. For this reason it is common practice to use computer simulation packages, similar to that used to produce Fig. 23, to optimize the cycle, having regard to the possible different thicknesses of the components being processed in the same run.

5.10 Performance, Productivity, and Economics

Autoclave processed prepreg is acknowledged to offer the best mechanical performance (stiffness and strength) of all composites systems. This is because a high V_f is achieved, with precise control of fiber architecture, good dimensional control, and high molding quality. The process is also very versatile in the variety of component forms that may be produced, and it is often possible to gain from parts consolidation, with consequent savings in assembly and inspection costs. The downside is that the feedstock is the most expensive format for all fiber types, capital equipment and/or labor costs are high, and the process cycle is very time consuming. It would be very difficult to achieve a production rate of more than 125 parts/annum from a single tool set, i.e., one part every two working days. In the context of the aerospace industry, this may be acceptable but this is less likely to be the case in other industries. Even within the aerospace industry, there is intense pressure to develop more cost-effective techniques that maintain structural efficiency and quality at acceptable levels. Foremost among the contenders are RTM and resin film infiltration (RFI) and also some possibilities with thermoplastics matrices. These are all discussed in subsequent sections.

6 OTHER PROCESSING OPTIONS FOR PREPREG

Although prepreg is mainly processed in the autoclave, there are some other options. One of the most straightforward is to use a vacuum bag, as in the autoclave process, but to cure in an oven or by application of radiant heat to the mold. A number of low-temperature curing resin systems are available, allowing cure at temperatures as low as 60°C. These systems are also widely used for the manufacture of tooling for autoclave and other processes. Another option, useful for very large moldings, e.g., hull moldings for yachts, is to construct a temporary enclosure around the mold, from tarpaulin or plastic sheet and heat with hot-air blowers. Prepreg may also be used alone or with other reinforcements for contact molding or press molding. Simple moderately curved shapes may be hot press molded using an elastomeric counter tool. For nearly flat panels a simple block of elastomer (e.g., a soft rubber) may be used, while for more complex shapes a tailored counter tool is used. These may be manufactured from castable polyurethane compounds.

7 RESIN FILM INFILTRATION

7.1 Basic Principles

The principle of resin film infiltration (RFI) has been used for many years, but the nomenclature RFI is comparatively recent. The concept is to use a dry reinforcement and to interleave it with resin, nominally in the form of a thin film. Under pressure and heat the resin viscosity is reduced so that it infiltrates through the reinforcement and ultimately cures. Processing conditions must be set so that the resin completely infiltrates the reinforcement and porosity is eliminated. The principle is illustrated in Fig. 24. Clearly more densely woven and thicker reinforcements will be less permeable and require a longer process window. The resin systems also need to be selected to give an adequate window of low viscosity before gelation. The advantages of this technique is that the dry reinforcements are a much cheaper feedstock than prepreg, the resin film may be accurately placed to ensure the correct fiber/resin proportion, and the infiltration distance is very short, just the thickness of a single layer of the reinforcement. This may be compared with the situation in resin transfer molding (RTM) where the infiltration lengths may be of the order of 1 m (Section 8).

7.2 RFI with Autoclave Cure

The tooling and general setup are similar to that used for prepreg. The reinforcement is typically lightweight woven or noncrimp fabric, usually of 200–600 g/m^2. The layers of reinforcement are laid on the mold and interlayered with the resin film. This is usually supplied on a paper backing similar to that used for prepreg. This is dispensed to give the required final V_f. Not all resin systems

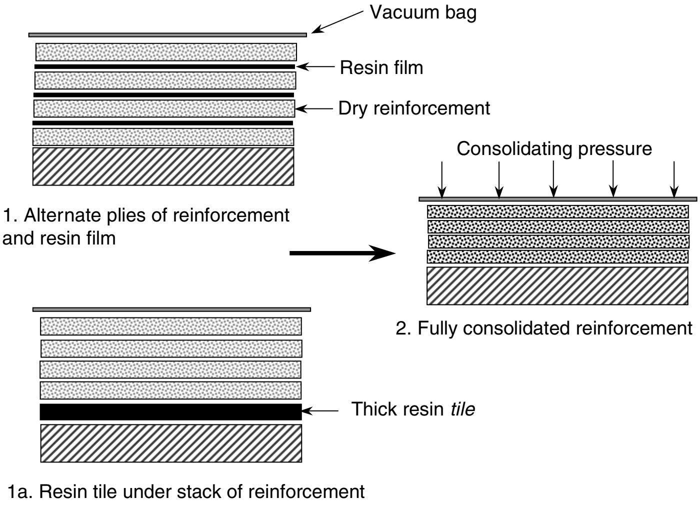


Fig. 24 An illustration of the principle of resin film infiltration (RFI). In 1 the reinforcement plies are alternated with resin film. With this arrangement the infiltration distance is only the thickness of a ply. The alternative system, 1a, uses a thicker resin layer (tile) placed under the whole laminate. The flow distance in this case is the whole thickness of the laminate. This system may be satisfactory for thinner laminates and when the resin melt viscosity is sufficiently low. Resin tile may be a more economic form than resin film.

are suitable for the production of film, and in some cases thin cast slabs of resin *(tiles)* are used. The completed lay-up is covered with release membrane, breather, and vacuum bag and processed in essentially the same manner as prepreg. The economic advantages over prepreg stem from the use of the cheaper fabric feedstock. The build rate is also faster than for prepreg due to the higher areal density of the fabrics used. The resin cost is high due to the need to manufacture it in film or tile form. The overall V_f achieved is likely to be somewhat lower than with prepreg, and there is some loss of flexibility in designing the laminate configuration. This results in slightly lower structural efficiency, but this is usually more than compensated by the lower cost. Productivity may be marginally better than with prepreg because of the faster build rate.

7.3 RFI with Press or Oven Cure

Instead of the autoclave the molding may be cured by vacuum bagging and oven cure. Clearly this does not allow as much consolidation pressure as in the autoclave, but very satisfactory results may be obtained with some resin/reinforcement combinations. Radiant or hot-air heating may also be used. Parts of simple shape and moderate size may also be hot press cured using two part matched tools or elastomer counter tools. There are considerable economic advantages with these methods compared with the autoclave process, although there is sometimes some performance loss due to lower V_f and higher porosity levels. Productivity can be significantly higher due to the elimination of the lengthy autoclave process.

7.4 Other RFI Options

The term RFI implies the use of a resin film, but this option is relatively expensive. A viable alternative is to apply the resin, as a liquid, by spraying onto each layer of reinforcement as it is placed on the tooling. The resin may be briefly preheated to lower its viscosity for spraying. It will then thicken as it is cooled by contact with the cold reinforcement. A novel development has been the introduction of semiimpregnated reinforcements. These materials consist of two layers of dry reinforcement with a layer of pasty resin sandwiched in between (see Fig. 4). This material has a number of advantages. It is dry to handle and may be easily cut to shape and placed in the mold. It also has a unique *dead* handling characteristic. It is much less springy than normal dry reinforcements, and, when draped over details on the tooling it tends to hold its shape. This facilitates lay-up. It can be manufactured from all conventional forms of reinforcement, random mat, woven cloth, and noncrimp fabric, and the additional cost is quite modest. It is manufactured in areal densities of up to 5000 g/m^2, which allow fast build rates, in E-glass fabrics. In combination with low-temperature cure resins, it is a very attractive for the manufacture of large moldings, using a vacuum bag with radiant or hot-air heating. This method has been used for the production of wind turbine blades of over 25 m length.

8 RESIN TRANSFER MOLDING

8.1 Basic Principles

Resin transfer molding (RTM) is a relatively new term for a process that has been used in various forms for many years and under various names. Essentially it involves infiltration of dry reinforcement in a closed mold. The variants depend on the type of tooling, the method for inducing the resin to infiltrate, and the resin cure temperature. The following are the main variants:

RIM	Reaction injection molding
RRIM	Reinforced reaction injection molding
SRIM	Structural reaction injection molding
RTM	Resin transfer molding
VARI	Vacuum-assisted resin injection
VARTM	Vacuum-assisted resin transfer molding
SCRIMP	Seeman composites infusion molding process

Some of these are discussed in detail in this and later sections. There follows a brief description of each.

1. *RIM.* This term is generally used for moldings manufactured by injecting, at high pressure, a reactive blend of precursor chemicals that rapidly cure. The classic system uses polyurethane chemistry and approximately equal proportions of an isocyanate and a polyol are reacted to form a polyurethane. The resin is not reinforced, so this is not a composite. The cured polyurethane may be formulated to be hard and rigid, a flexible elastomer, or either rigid or flexible foam. Cure time is of the order of 1 min and molds are unheated, but there is some heating due to the cure exotherm. Alternative resins may be based on polyester or polyamide chemistry, but these not widely used. While RIM is not a composite system, similar chemistry and process principles may be used for composites manufacture.

2. *RRIM.* This is a reinforced version of RIM. The same polyurethane chemistry is used but very short chopped, or milled, glass fibers are dispersed in the polyol component. The molding procedure is similar to that for RIM, but the cured material will be reinforced with the glass fibers. The fibers must be very short so that they can be pumped through the injection system, and for similar reasons the fiber loading must be quite low. Fibers cannot be dispersed in the isocyanate component because they initiate chemical reactions that would reduce the pot life of the isocyanate and interfere with the cure process. The total V_f in the final molding is likely to be less than 0.15, and this, combined with the very short length, means that the degree of property enhancement is low. However, the reinforcement is sufficient to improve the dimensional stability of the moldings and is most useful for soft components: e.g., automobile interior moldings and front-end moldings designed to protect pedestrians in the event of collision.

3. *SRIM.* This a true RTM process. Similar urethane technology is used but dry reinforcement is placed in the mold and, after mold closure, the resin is injected. It infiltrates the reinforcement and cures in about 1 min. A schematic of the basic principle is shown in Fig. 25. This process is widely used for the manufacture of automobile and truck dashboards. The resin formulation is designed to provide some protection to the occupants in case of accident. Rigid

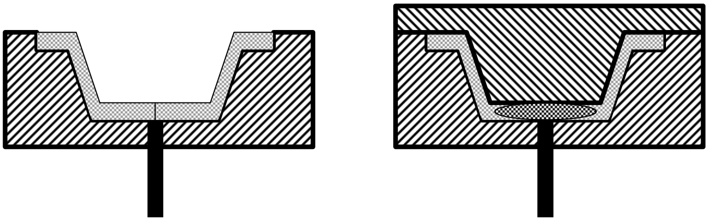


Fig. 25 Illustrates the principle of both SRIM and RTM. The dry reinforcement, usually as a preform, is placed in the open mold. The mold is then closed and the liquid resin injected into the cavity to fully infiltrate the reinforcement. The difference between the two processes is that in SRIM a rapidly injected polyurethane resin curing in 1–2 min is normally used. In RTM, conventional UPE and epoxy resins with substantially longer processing times are used.

resin formulations may also be used. The reinforcement is most often a fairly open random chopped strand. This provides high permeability so that the resin may be rapidly injected. If the permeability is too low, pressure gradients may build and distort the reinforcement. Fiber loadings of 0.1–0.25 are common, and this provides a useful level of property enhancement, especially dimensional stability at slightly elevated temperatures.

4. *RTM.* This is similar in principle to SRIM. The reinforcement is preplaced in the mold and the resin injected after mold closure. In RTM a much higher V_f may be achieved and consequently the lower permeability of the reinforcement means that much slower infiltration over a longer period is the rule. Lower pressures allow for use of less robust and cheaper tooling. Cure times are seldom less than 20 min and often extend to several hours. Variants involve the type of tooling, infiltration pressure, and cure temperature. These are discussed at length below.

5. *VARI.* Has been used for many years. It is simply RTM in which a vacuum is applied to the cavity so that the resin is forced into the mold by atmospheric pressure. Preevacuating the cavity helps prevent air being entrapped in the molding, a hazard when only a positive pressure is applied.

6. *VARTM.* This is a more recent nomenclature for VARI; it is identical. It is probably a reinvention.

7. *SCRIMP.* This is also a recent term for a essentially well-established variant of RTM. SCRIMP is a patented proprietary process, but some aspects of the principle are used in other nonproprietary processes. This uses a single-sided tool with a vacuum bag. A vacuum drawn between the bag and the tool induces resin flow into the reinforcement by atmospheric pressure. The novelty of the SCRIMP process is the use of a sacrificial mesh, illustrated in Fig. 26, between the lay-up and the bag. This acts as a permeable feeder ply, allowing resin to flow rapidly over the entire area of the molding and then to flow transversely through the thickness of the reinforcement to complete the infiltration. Earlier practice was to incorporate layers of CSM on top of or between layers of denser reinforcement to accomplish similar ends.

8.2 Basic Process Details

The principal variables of the process are the type of tooling, the method of resin transfer, and the cure temperature. There is also the question of size and

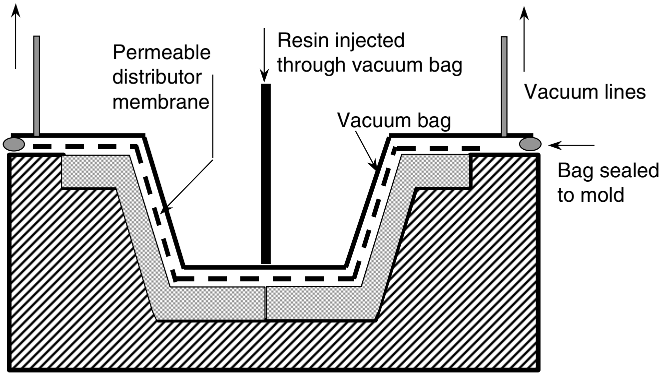


Fig. 26 In the SCRIMP process, a single-sided tool is used and a permeable distributor membrane is incorporated. The membrane allows the resin to flow rapidly over the entire surface of the molding, so that the infiltration distance is only the laminate thickness. This results in much faster infiltration than conventional RTM and is especially suited to moldings of large surface area.

required production rate. RTM is used for manufacture of parts ranging in weight from less than 1 kg to over 1 ton and this has a profound effect on the type of tooling that is viable and to both transfer pressure and cure temperature. An important feature of the process is that the mold is sealed and consequently vapor emissions are much reduced compared with contact molding.

Tooling may be composite or metallic. Most large molds are composite, smaller tools may be composite for short runs but metal for long runs. If high pressure or hot cure is used, it is generally necessary to use metal tooling. Conversely high pressure is not appropriate for very large parts due to the need for very robust tooling.

Most RTM is carried out with matched tooling in which the cavity is precisely defined. In the case of large molds the two halves may be bolted or clamped together for the molding operation. Smaller molds may be mechanically opened and closed, e.g., in a press. The SCRIMP process and variants use one-sided tooling sealed with a vacuum bag. This is much cheaper than matched tooling but does not result in a fair surface on both sides of the molding. Furthermore, pressure infiltration is not an option, only vacuum may be used. This can lead to higher levels of porosity than if pressure is used. It can be difficult to efficiently infiltrate moldings of high surface area, which are made up from high V_f reinforcements, owing to the combination of long infiltration paths and low in-plane permeability. This problem is overcome in the SCRIMP process by using a high-permeability mesh ply, under the vacuum bag covering the whole molding area. A permeable peel ply is placed between this mesh and the top plies of the lay-up. This may be a layer of PTFE-treated glass cloth, similar to that used under the resin bleed ply in autoclave molding. The resin infiltrates rapidly through the mesh and then passes through the peel ply and into the lay-up. Although the permeability of these layers may be quite low, the infiltration path is very short, just the thickness of the molding, so that complete infiltration is attained in a reasonable time. After cure the peel ply and mesh are stripped from the surface of the molding and discarded.

The transfer of resin into the tool may be assisted by vacuum, gravity, or positive pressure or a combination of these. Vacuum is simple to engineer, and, provided the tooling is sufficiently robust, there need be no problems due to mold distortion. The resin is forced into the mold by atmospheric pressure. Preevacuating the mold before opening the resin port removes most of the air and vapor from the mold interior and helps prevent noninfiltrated areas. It is often necessary to restrict the flow of resin to prevent *racetracking*. Streams of resin flow through the mold sealing off pockets of reinforcement that then never fill (Fig. 27). The general strategy is to admit the resin at the bottom of the mold and to vent around the upper periphery. The riser tubes lead through resin traps to a vacuum manifold. If the resin flow is correctly throttled, the mold fills uniformly from the bottom, and filling is indicated by the appearance of resin in the transparent risers. These are then shut off, pinched, to stop further resin flow and prevent resin getting into the vacuum system. Further assistance may be provided by arranging a positive head of resin above the inlet port and also above the highest point of the mold so that there is gravity assistance. Low-pressure assistance may be arranged by use of a pressure pot, where the resin container is subjected to an overpressure of gas, usually nitrogen, at pressures of up to about 5 bars. This may be used in conjunction with preevacuation, but once all the risers have been pinched off a positive hydrostatic pressure is generated within the mold cavity. This ensures complete filling and will reduce porosity by collapsing bubbles and forcing vapors into solution in the resin. Higher pressures may be achieved by the use of a positive displacement resin pump. Pressures of up to 10 bars are commonly used. The pump generates a constant flowrate and the pressure does not build up until the mold is full. Again preevacuation is beneficial.

The next consideration is cure temperature. Ambient curing systems are widely used, and these must be formulated so that the resin viscosity remains

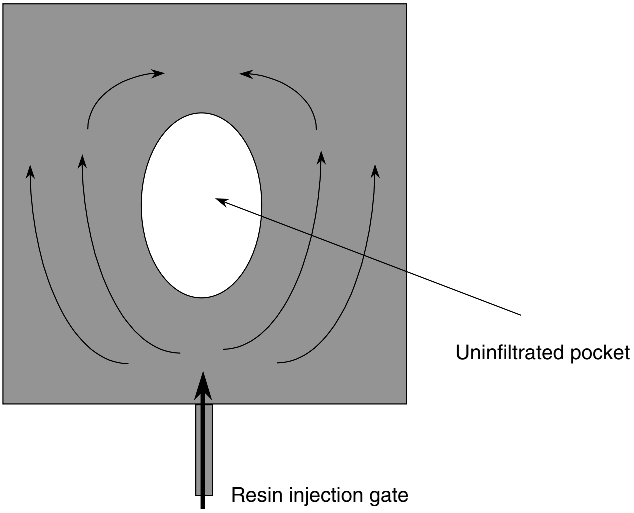


Fig. 27 Uncontrolled flow in RTM processes can lead to the formation of unwetted pockets as illustrated in this schematic. These may be avoided by proper positioning of the inlet gates and by control of the flowrate.

sufficiently low, typically <100 Pa/s, for the time interval necessary for full infiltration to occur. The consequence of this is that gel time will normally need to be at least double the infiltration time and that the time to demoulding will be of the order of 5 times the infiltration time. The cure process may be speeded up by the application of heat after the mold has been filled. Small molds may be placed in an oven and large molds may be heated by radiant heaters or blown hot air. With composite tooling the maximum cure temperature is unlikely to be much more than 100°C. If metal tools are used and buried heaters are used, the molds may be heated directly to higher cure temperatures, typically 120–180°C. Electric cartridge heaters, circulating hot oil, and steam heating can be successfully used for mold heating. In general with thermoset resins the parts may be ejected hot. To avoid having to cool the molds between cycles, it is advantageous to keep them continuously at the cure temperature. The reinforcement is placed into the hot, open mold, and after closure the resin is injected. The danger with this system, especially if there are long flow paths, is that the resin should gel before infiltration is complete. A further strategy is to preheat the resin to a temperature lower than the cure temperature, e.g., 50–70°C. This lowers its viscosity allowing faster infiltration. Clearly great care must be exercised to formulate the resin and control the temperatures so that full infiltration is ensured and cure is achieved in the shortest reasonable time, so that productivity is maximized.

8.3 Low-Pressure, Ambient Cure RTM

This is the longest established version of the process. In most cases two-part matched tooling is used. For short runs composite molds are used, but the supporting structure needs to be more robust than for ordinary contact molding. According to the size and complexity of the part, the reinforcement is either laid directly into the mold or a preform is separately made up and simply placed in the open mold. The mold is then closed and the resin infiltrated into the reinforcement. There are several strategies for resin infiltration: The attraction of the system is that very large moldings can be economically made this way with relatively cheap and simple tooling. Using matched tools means that dimensions, especially thickness, is more precisely defined and all surfaces are fair. A gel coat may be used to enhance surface finish, or color, if required in the same way as with contact molding. In large moldings, e.g., the hull of a 10-m sailing yacht, the reinforcement is laid in the mold. This may take several days. A variety of inserts, such as foamed sheet to form sandwich sections or timber pads for reinforcing mounting points, may be incorporated into the reinforcement. Pressure infiltration is not usually an option with large moldings but a combination of preevacuation with gravity-assisted infiltration, i.e., maintaining a hydrostatic head of resin, can be used.

For smaller moldings it is common to use preforms, as this reduces the length of time that the molds are being prepared. Composite tooling is adequate for short runs, but metal is preferred for larger numbers of moldings. The tools may be mounted in a press, which facilitates opening and closing and also enables the injection system to be permanently connected. Higher pressures may be used and, since resin flow paths will be much shorter, infiltration time will be faster, so that faster curing resins may be used with, consequently faster, mold turnaround time. A combination of low pressure, 1–5 bars, with vacuum is typical.

When positive pressure is used, the risers over the vent ports may be closed once the resin has infiltrated, so that a positive hydrostatic pressure is generated in the cavity. This ensures complete filling and reduces porosity by collapsing air or vapor bubbles.

8.4 High-Pressure RTM

This is applicable to fairly small components, area $<2\ \text{m}^2$. In this case metal tooling is used and hot cure is normal. The molds are normally operated in a press, facilitating opening and closing and providing the means to hold the tool closed against the injection pressure. Pressures of 5–10 bars are common, and the mold cavity is preevacuated. Preforms are the preferred form of reinforcement when cycle times of $\approx$15 min are possible with small components.

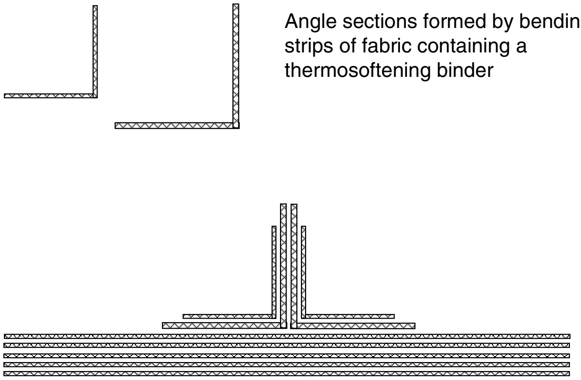
8.5 Manufacture of Preforms for RTM

Many laminating processes may be speeded up by the use of preforms. This enables the whole lay-up process to be completed before the mold is loaded. Effectively preform manufacture and molding can be carried out concurrently. This can often double the rate of production from a molding station.

The main objectives in preform manufacture are to assemble the necessary layers of reinforcement and then to consolidate them sufficiently so that they may be placed in the tooling. The problem is excessive bulk, which might obstruct mold closure. The consolidation is effected by precompressing and the use of binders, which may be heat set. The choice of type and proportion of binder is vital for successful preform manufacture. Many reinforcements are available already treated with a suitable binder system. In other cases it may be necessary to apply binder during preform manufacture, usually by spray or as a powder. The binders may consist of a similar resin to that used for the matrix or either a thermoset or thermoplastic that is compatible with the system being used. The binders are either B-staged thermosets (solid or pasty at ambient temperature) or thermoplastics with softening temperatures of about 100°C. When the reinforcement is preheated and then compressed and allowed to cool under pressure, the binders melt and then freeze, stabilizing the preform in the compressed state. The sequence of operations is to lay up the required reinforcements in an auxiliary half mold. This is then heated by radiant heaters, blown hot air, or sometimes microwave to soften the binder, before being compressed, either using a vacuum bag or between cooled shaped tooling. The principle for building up details using this technique is shown in Fig. 28.

Simple random preforms are manufactured by blowing chopped strand onto a mesh shape on a vacuum box. This is similar to the spray-up process described in Section 3. When the preform is of the required thickness (the weight is usually checked), it is consolidated as described above. The periphery may also be trimmed after consolidation. The finished preforms are sufficiently robust to be handled with care. They are delivered to the molding station for final processing.

More complex preforms may be constructed from random mat, woven, and noncrimp fabrics and from braided or knitted materials. In some cases it is necessary to heat set individual plies before they are assembled on the preform (e.g., to form stiffening ribs—Fig. 28). Such preforms may require several hours



Angle sections formed by bending strips of fabric containing a thermosoftening binder

Flat reinforcement and preformed angle sections assembled to form a stiffening rib. Heat tacked into place.

Fig. 28 Complex detail may be built into a preform by using thermoformable reinforcements. These incorporate a thermoplastic binder so that they may be heat-set before assembly to form the preform. This is illustrated by a blade stiffener built up from thermoformed tapes.

of hand work to manufacture and a sufficient number of parallel preform manufacture cells must be set up to match the molding rate of the final tool.

Another strategy is to use an auxiliary tool (or a number of such tools) on which the preform is assembled and preconsolidated. The whole auxiliary tool complete with preform may then be loaded into the main tool. Effectively the auxiliary tool is a thin shell that accurately matches the bottom half of the main tooling. It needs to be much less robust than the main tooling, just sufficient to allow it to be handled between the preform assembly cell and the molding station. It is a much more economical option than having duplicate molds. The auxiliary shells may be composite or even formed sheet metal. They may be regarded as disposable, being used to produce a short run of moldings before being replaced, while the wear and tear on the main tool is reduced and its useful life extended. A schematic layout using auxiliary tooling is illustrated in Fig. 29.

9 FILAMENT WINDING AND TOW PLACEMENT

9.1 General Concepts

These are two related processing concepts in which continuous fiber roving is directly formed into a component. This is attractive because continuous roving is usually the cheapest format of the reinforcement fiber, as there is no downstream process cost after fiber manufacture. The basic principle is that dry fiber rovings are impregnated with resin and laid down on a tool or former in the required sequence to generate the desired reinforcement configuration. The use of single rovings facilitates impregnation by the matrix resin and also the continuous uniaxial fiber arrangement gives the possibility of achieving very high fiber fraction in the final product; $V_f = 0.8$ is typical. The rovings are maintained very straight and in a precise lay-up. All these features contribute to the realization of excellent mechanical properties and structural efficiency. Both pro-

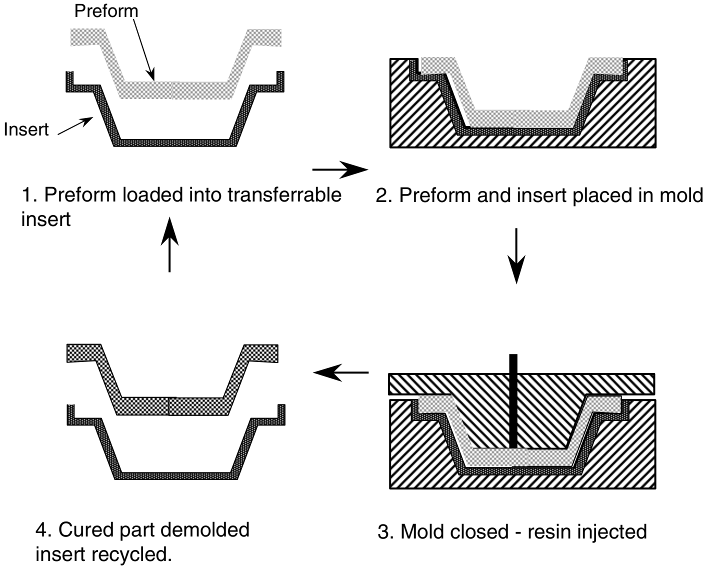


Fig. 29 Productivity in the RTM process may be enhanced by use of a system of transferable mold inserts. These are auxiliary tools, usually thin sheet metal construction. The preforms are built up on these inserts, which are finally placed into the RTM molds. Several inserts may be rotated, so that preform assembly and demolding may be carried out concurrently with molding.

cesses utilize fully automatic techniques that are usually computer controlled. Labor requirements are low but the rate of build also tends to be quite low.

9.2 Filament Winding

The principle here is that the fiber roving is laid onto a rotating mandrel by a dispensing head, which may simply reciprocate or execute more complex motion. In the simplest version a cylindrical mandrel is rotated and the dispensing head traverses forward and backward parallel to the axis of the mandrel. This results in a simple structure laid at angles of $+\theta$ and $-\theta$ to the mandrel axis (Fig. 30). This produces a tubular structure. The angle of the lay-up is controlled by the relative speeds of the rotation and the traverse, and it is convenient to wind at angles from near 90°, the hoop direction, to about 25°. Clearly it is not easy to wind close to the axial direction with this arrangement, but alternative machine configurations allow this to be done, e.g., the race track winder (Fig. 31). A number of other configurations are also possible so that virtually any desired configuration can be attained on axisymmetric shapes. By incorporating extra degrees of freedom in the winding head, nonaxisymmetric shapes may also be wound. However, the main output of commercial filament winders are cylindrical tubes and pressure vessels. In the case of tubes there is a transition region at each end when the traverse reverses. This is a ring of thicker section where the angle changes from $+\theta$ to $-\theta$ and vice versa. This is usually trimmed off. For pressure vessels a cylindrical section blends into hemispherical ends. This enables the winding to be carried over the end as the traverse changes direction. This provides an ideal fiber configuration for pressure applications. The cylin-

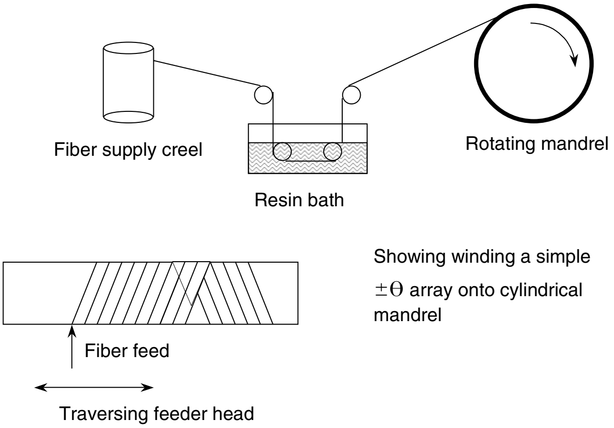


Fig. 30 Basic filament winding process. Impregnated fiber tows are wound onto a rotating mandrel. Control of rotation and traverse speeds determines the angle of wind. A $\pm\theta$ pattern is built up as the traverse direction is alternated. This results in a basket weave structure.

drical portion is wound at ±55°, which gives twice the strength in the hoop direction compared with the axial, an ideal balance. The fibers are wound uniformly in all directions over the hemispherical ends giving isotropic properties. An alternative is to wind a spherical vessel. To wind hollow pressure vessels it is necessary to devise a means of removing the mandrel. Various techniques have been adopted: inflatable mandrels, plaster and salt mandrels, which are leached out with hot water after the part has been cured, and expanded polystyrene, which can be collapsed by heating or use of hydrocarbon solvents.

Filament winding is ideally suited to the manufacture of axisymmetric hollow shapes, but, as indicated above, some nonaxisymmetric shapes can also be formed. There are some restrictions. The winding path needs to comply quite

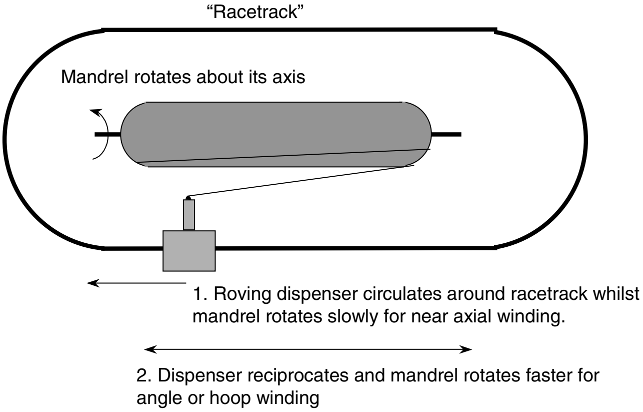


Fig. 31 Setup illustrated in Fig. 30 is not suitable for laying tow oriented closer than about 20° to the mandrel axis. Zero winding angles may be laid with a race-track machine. This is suitable for axial, hoop, and angled windings according to the relative speeds of the race-track dispenser and the mandrel rotation.

closely to a geodesic path (great circle path) on the curved surface, otherwise the winding tension will lead to lateral slippage of the wound rovings. Friction may allow some small deviations from the ideal path. Another restriction is that it is generally not possible to wind into concave surfaces. These restrictions do not apply to winding thermoplastic matrix systems, which are effectively welded into place as laid (see Section 9.3).

Filament winding usually uses continuous dry rovings that are impregnated with resin by passing through a trough of liquid resin immediately prior to deposition. The resin content is controlled by the resin viscosity and by passing the wet tow through a calibrated *eye,* which squeezes out any excess resin. This is a simple and effective technique. After winding, the resin is allowed to cure either at room temperature or at elevated temperature. An oven or radiant heater may be used. It is sometimes desirable to rotate the mandrel during cure to prevent any excess resin running to the bottom of the winding.

As the roving is laid down on the mandrel, it will be in the form of a flat ribbon, and to obtain a uniform winding it is necessary that complete coverage be obtained with a integral number of ribbons, without overlap or gaps. It is common practice to combine several rovings to form a wider ribbon. This ribbon is then wound as an open helix along the length of the mandrel. The next run will be in the reverse direction and will consist of a helix of opposite angle. It must be arranged that the width of the tape, w, is related to the circumference of the mandrel, πd, so that $nw/\sin\theta = \pi d$ (Figs. 32 and 33). The number of double runs necessary for complete coverage is then equal to n. It is also necessary that the start point for each run be positioned so that each ribbon is perfectly butted to its neighbors. The ribbon width must be chosen so that a logical sequence of positions may be realized. For instance if $n = 7$, there must be 7 start points around the circumference at each end of the mandrel. The sequence for winding may then be 1, 2, 3, 4, 5, 6, 7; 1, 3, 5, 7, 2, 4, 6; or 1, 4, 7, 3, 6, 2, 5. Each of these results in a slightly different variation of the basket weave structure that characterizes filament winding.

Filament winding may also be carried out using preimpregnated tows or with prepreg tape. It is also possible to wind fibers preimpregnated with thermoplastic resins. In this case local heat is applied to melt the thermoplastic, and pressure

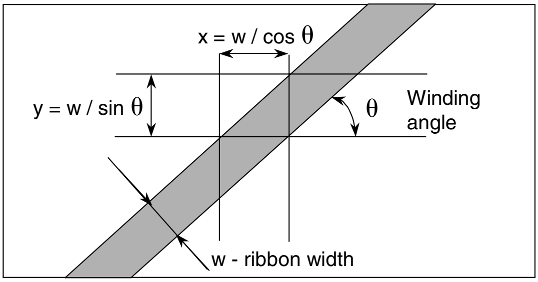
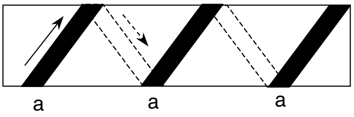
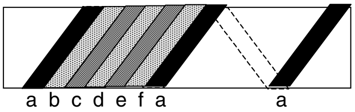


Fig. 32 Illustrates the concept of ribbon width. The ribbon is normally built up by laying several impregnated tows simultaneously. The axial and hoop coverage are then simple functions of the winding angle.



1. Single pass produces an open helix



2. Ribbon width must be arranged so that an integral number of passes produces a complete cover - 6 in this illustration.

Fig. 33 Illustrates how a full cover is achieved by laying a number of ribbons successively so that they each butt up to each other. The ribbon width and the start position for each pass must be carefully synchronized to achieve this effect.

is applied to consolidate at the point of application (Fig. 34). This is essentially welding the tape or roving into position as it is laid.

Tubular components are widely used for transport of fluids at moderate pressures and also for drive shafts. In the latter case the configuration is modified. Layers at ±45° are used to carry torsion loads, and others at 0° for bending. Machinery for simple filament winding is relatively inexpensive, it can be operated automatically with manual attention only necessary to change mandrels when a winding run is completed. The range of sizes is wide, varying from less than 10 mm diameter up to more than 3 m. Lengths are limited by the length of mandrel that can be supported without undue sag. Machines have been de-

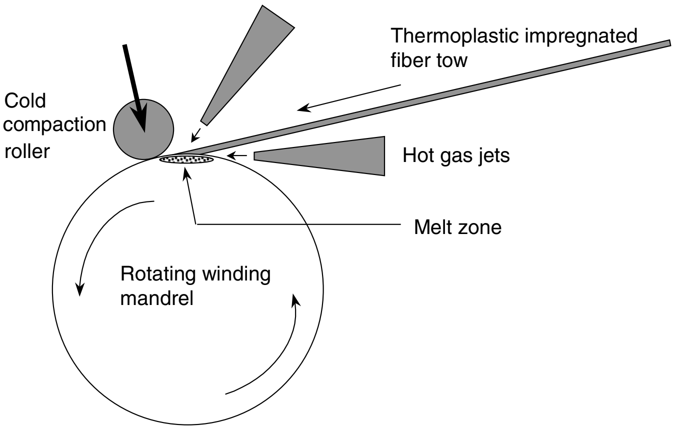


Fig. 34 Thermoplastics impregnated tows may be filament wound by thermally *welding* them into place at the point of application. The principle is illustrated in the figure. This technique can also be adapted for tow placement technologies.

veloped, with reciprocating mandrels and *in situ* cure, that allow for the manufacture of continuous piping.

9.3 Tow Placement

Tow placement technology is a variant of filament winding where impregnated tows (rovings) are precisely placed onto the mold surface using a robotic tow placement head. The basic technique is to place the tows into/onto a stationary mold, which may be flat or have complex curvatures. Further part shape possibilities may be generated by moving the mold as well as the placement head. A standard tow placement machine is similar in many respects to a tape laying machine with the placement head operating from a gantry moving over a flat bed on which the tool is mounted. Other machines are essentially robots that bear the tow placement head and can be manipulated with 3–7 degrees of freedom according to the complexity of the surfaces to be generated.

The tow or roving is normally fed dry into the placement head, where it is impregnated with resin prior to being placed in position. Pressure is applied at the point of placement to bond the laid tow to the previously laid layers or to the mold surface if it is the first layer. The alternative is to use preimpregnated tow, which is supplied in spools and stored at low temperature, like prepreg. It is also important that the impregnated tow have a degree of tack, so that it remains in place once laid. One strategy is to warm the impregnated tow immediately prior to placement; this softens the resin. After placement it cools, the resin viscosity increases sharply, and it will be effectively stuck in place. This strategy is also the basis for tow placement using preimpregnated thermoplastic matrix tows. In thermoset systems the completed laminates are then cured using conventional autoclave or vacuum bag and oven cure technologies.

Tow placement is a more versatile technique in terms of shapes that may be generated than either filament winding or tape laying. There is no requirement that the placement head follow a geodesic path or that the path needs to be straight. Tows can be effectively laid into concave surfaces. The normal strategy is to lay the tows in layers to build up the required laminate configuration. The tows are cut at the end of each traverse and the head reset for the next run. The limitations of tow placement are the high acquisition costs of the placement equipment and a relatively low rate of production. The maximum placement speed is currently of the order of 1 m/s. If the tow weight were 2500 tex, a heavy tow, this translates to a placement rate of about 9 kg/h. This does not allow for inevitable losses due to the need to manipulate the head each time a run is completed. In spite of these disadvantages, tow placement is seen as one of the more cost-effective techniques for laying up high-performance structures for aerospace and similarly performance-dominated applications.

10 PULTRUSION

10.1 Principle

This is a versatile process for the manufacture of prismatic sections. In this respect it differs from most composites technology in that a semifinished product rather than a component is manufactured. The concept is very simple; continuous reinforcement is impregnated with liquid resin and then drawn through a long,

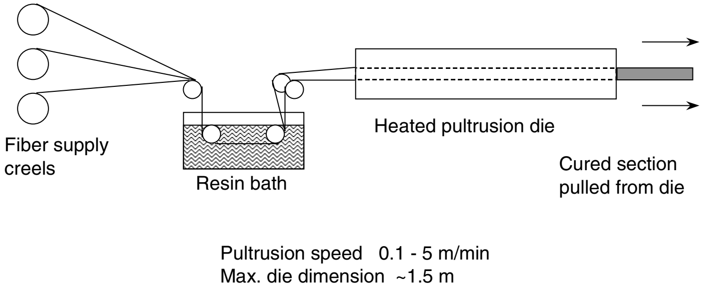


Fig. 35 In the basic pultrusion process, resin impregnated tows are pulled through a heated die, from which they emerge as fully cured solid sections.

heated, shaped die. During its passage through the die, the resin is cured and a solid section emerges. The continuous emerging section is then cut off into convenient lengths for transportation and conversion into the finished product. Slender sections may be coiled so that very long lengths can be manufactured. This principle is illustrated in Figs. 35 and 36. The traction to pull the material through the die is applied to the emerging cured section, either a caterpillar or more usually a pair of reciprocating pullers are used. These are operated by a chain drive in a manner analogous to "hand-over-hand" pulling a rope. One puller is clamped to the pultrusion and engages in the drive chain. It pulls the product for a short distance, e.g., 1–2 m, while the second puller is returned to its start position. Then the second puller is engaged and the first released and returned. The pullers are easily adapted to different pultruded profiles by simply

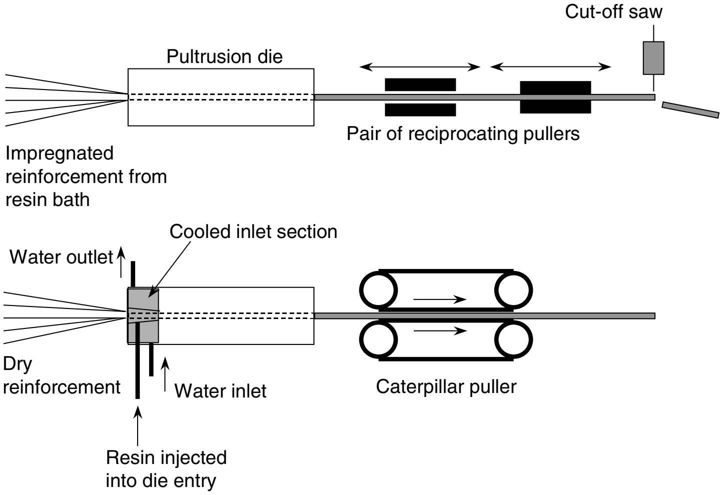


Fig. 36 Two alternative techniques for pulling pultruded sections are illustrated. The reciprocating puller is more easily adapted to different section shapes and is generally the more versatile. Also illustrated is the alternative resin injection technology, where resin is injected into a cooled entry section of the die, rather than the reinforcement being impregnated by passing through a bath of resin.

changing the shaped clamping pads. Caterpillar traction is suitable for handling flat products but is generally not as versatile as the reciprocating system. The reinforcement may be impregnated by passing it through a bath of resin, as with filament winding, or by injecting resin into the entry portion of the die. These points are all illustrated in Figs. 35–38.

Solid and hollow sections may be manufactured and virtually any external profile that can be contained in a window approximately 2 × 1 m. Pultrusion speeds are quite low, seldom more than 2–3 m/min. The speed is dictated by the length of the die and the necessary cure time, which is temperature dependent. In practice die temperature and pulling rate are adjusted so that the section is sufficiently cured when it emerges from the die. The process may be speeded up by preheating the charge as it enters the die or by partly curing in the die and then postcuring. There are shape restrictions, which are discussed below. There are also several options in choice of reinforcement and some possibilities for making curved sections and accommodating section changes along the length of the product. There is considerable interest in pultruding composites with thermoplastic matrices. In this case the reinforcement must either be preimpregnated or consist of one of the commingled forms, the resin in the form of fibers or strips of tape, interspersed with the reinforcement fibers. The charge must be heated to melt the matrix before or during entry into the first section of the die, which is hot. The charge then passes through a hot consolidation stage and finally through a cooled exit stage so that the matrix has solidified by the exit point. This is a developing technology and is not yet standard practice.

10.2 Reinforcement Options for Pultrusion

The basic requirement is that the reinforcement is continuous or at least in sufficiently long lengths that batches may be joined to maintain the continuous operation of the pultruder. The most obvious feedstocks are continuous rovings. The logistics of the process warrant some consideration. Individual packages (cheeses) of roving are generally supplied in weights of up to ≈25 kg. For a typical roving density of 2500 tex, this translates to a continuous length of 10 km, sufficient to keep the process running for about 100 h. One E-glass roving of 2500 tex corresponds to approximately 1 mm^2 cross-sectional area when molded. Thus, to pultrude a hollow section 100 mm^2 and of 3 mm wall thickness, i.e., cross-sectional area of approximately 1200 mm^2, 1200 individual roving packages, or *creels,* would need to be set up to feed into the die. Setting up the machine is therefore a significant logistical exercise. But, having set up the process, 10 km of the section could be manufactured in a single campaign, with very little supervision being required beyond maintaining the flow of resin and disposing of the product. One problem with using just roving as feedstock is that the product is composed entirely of uniaxial fibers. This may be acceptable for a limited range of products but some cross reinforcement is usually required.

Alternative forms of feedstock are tapes of any of the standard sheet forms of reinforcements. Chopped strand and continuous random mats are widely used together with woven and braided fabrics. These are invariably prepared as rolls of tape, trimmed to widths to suit the part being manufactured. These may be folded as they enter the die to form angle, channel, Tee and H sections, ribbed tubes, and a wide range of special sections, as illustrated in Fig. 37. Typical cross sections for pultruded products are shown in Fig. 38. Pultrusions may be

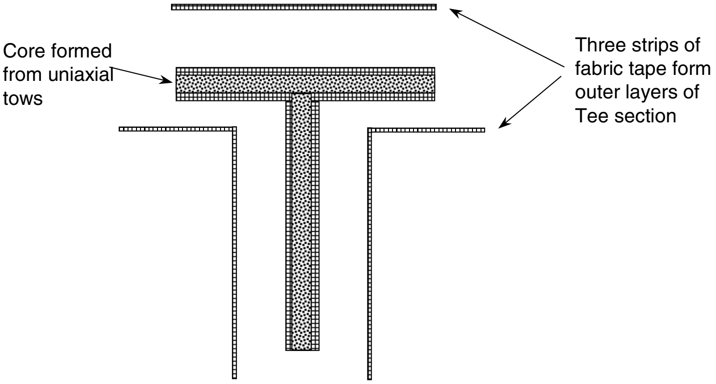


Fig. 37 Pultruded sections may be assembled from a variety of tow and tape reinforcements. This is illustrated for a Tee section formed from uniaxial tows in the core and three fabric tapes. These could be of either woven fabric or random mat.

manufactured in a similar range shapes as extrusions in aluminum alloys, and they are designed, in as far as is possible, so that they merely need to be cut to length to form the final component. The main principle for design of a section is to avoid having a wide range of thicknesses in a section, as this leads to difficulties in achieving uniform cure. Wall thicknesses of less than 1 mm are generally avoided, and simple profiles on the inside of hollow sections are preferred.

10.3 Process Variations

As noted in Section 10.1, for thermoset matrices the speed of the process is dictated by the need to adequately cure the resin by the time the product exits the die. Higher speeds may be achieved by operating the die at higher temper-

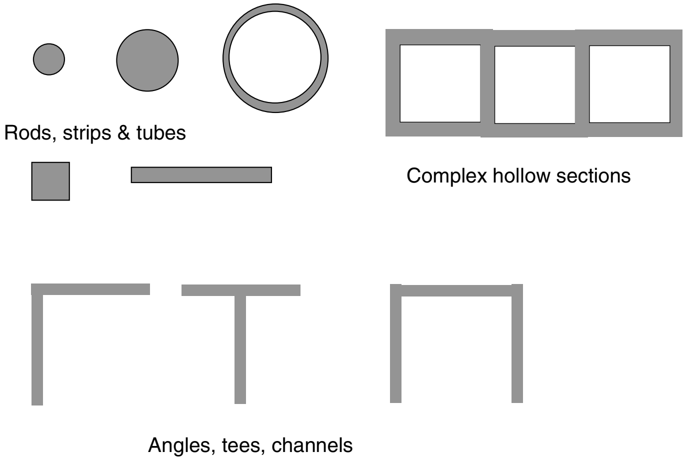


Fig. 38 Selection of section shapes suitable for pultrusion. The hollow sections are formed using a blind mandrel. There is no requirement for draft (taper) on the flanges of Tees and angle sections and corners can be quite sharp.

atures or by use of longer dies, so that the residence time is adequate to ensure cure. In practice these options are of limited use as the cure profile of most resins becomes unmanageable, due to excessive exotherm, if a temperature higher than the normal range cure temperatures is used. Also in longer dies the friction is greater and the pull traction needs to be higher. Heat transfer from the hot die to the charge is inefficient due to the low thermal conductivity of the charge. So, it is advantageous to preheat the charge, i.e., resin and reinforcement, at the point of entry into the die. This can be effected by microwave or dielectric heating according to the characteristics of the resin and reinforcement. Vinyl-ester and UPE resins can be heated by both methods, glass reinforcement is inert and does not interfere, but carbon is an electrical conductor and may interfere with these processes. Induction heating has been used with carbon but is not established practice because the vast majority of commercial pultrusions are made with glass reinforcement. Preheating must be done before the charge enters the metal die.

Most pultrusions consist of simple prismatic sections produced from passage through a single die. However, the linear nature of the pultrusion process makes it adaptable to multistage processing. For example, a tubular section could be formed from uniaxial glass rovings, then overwrapped using a pair of winding wheels to produce a $\pm\theta$ outer layer, before passing through the curing die (Fig. 39). It is also possible to produce curved pultruded sections by using a reciprocating die in the form of an arc of a circle. A related process, termed Pulforming, allows for the manufacture of parts with nonuniform cross section, by replacing the die by a series of clamshell molds carried by a pair of caterpillar conveyers.

10.4 Summary

Pultrusion is a versatile process and, although limited to the production of fairly simple cross-section prismatic shapes, it can be highly cost effective. It is most suited to the production of long runs of standardized sections. The capital costs

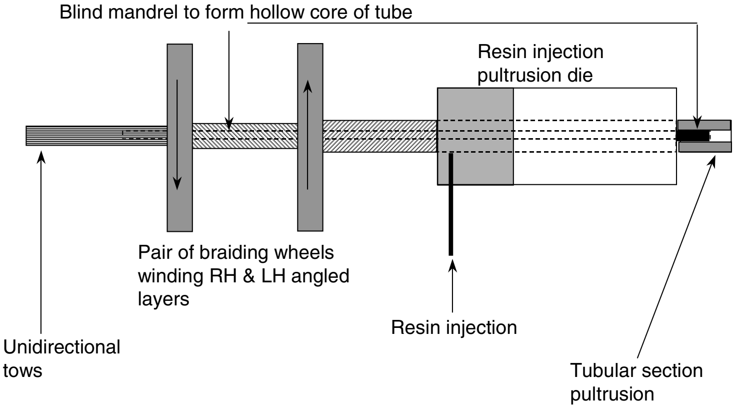


Fig. 39 Further possibilities are to filament wind, using braiding wheels, onto core sections formed from uniaxial or fabric reinforcements. The linear nature of the process allows for any number of operations before the section enters the die.

of equipment and tooling are relatively modest and the equipment, once set up, will run with minimal supervision. A number of construction systems have been developed that are based on the use of pultruded sections, often assembled by interlocking. These are widely used for the construction of corrosion-resistant industrial walkways, and one system has been developed for construction of bridges and associated structures.

11 CONTINUOUS LAMINATION

This is a specialized operation for the production of flat and profiled sheet materials, mainly transparent or translucent panels for the construction industry. Rolls of reinforcement, usually CSM or CRM, are fed through a series of rolls where liquid resin is incorporated. The impregnated material then passes through a curing section, which is typically a heated double-belt press. The material is cured during passage through the press, and the continuous section is cut to the required lengths by a flying saw or shears. If transparent sheeting is required, careful control of the choice of resin and reinforcement, especially the choice of binder on the reinforcement, is necessary, so that refractive indices of the glass and cured resin match and the porosity content is low. There is a danger that weathering in service results in disbonding of the resin/glass interfaces, which then reveal the fiber bundles and lower the transparency.

12 SHEET AND BULK MOLDING COMPOUNDS

12.1 General Principles

Sheet molding compound (SMC) and bulk molding compound (BMC) [also dough molding compound (DMC) in Europe] are both precompounded reinforcements intended for fast processing by compression, transfer, and injection molding. As its name implies, SMC is supplied in sheet form; usually in the form of rolls, typically 1–2 m wide and about 5 mm thick. Bulk molding compound is similar in constitution but supplied in bulk form, usually as a thick "rope," 20–50 mm diameter. The compounds consist of reinforcing fibers, almost always E-glass, and usually random discontinuous, fillers, and the resin, which is usually either UPE or vinyl-ester. The resins are specially formulated with a thickening agent so that they thicken to a stiff pasty consistency over a period of maturation of about 72 h after the initial compounding operation. This renders the compounds dry, nonsticky, and easy to handle. The SMC has a leathery consistency and may be cut with shears or blanked into shapes.

The compounds are cured by the application of heat and pressure. Initially the heat softens the resin so that the compound may be consolidated and molded into the die cavity by the applied pressure. The continued heating cures the resin. The whole process cycle may be completed in 2–20 min according to the section thickness and precise formulation.

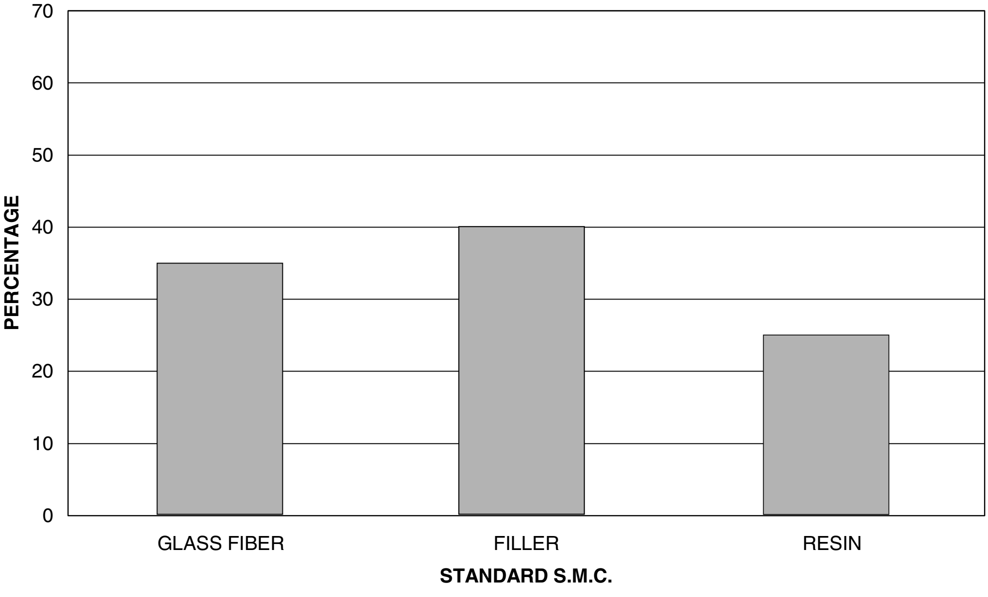
12.2 Sheet Molding Compounds

Standard SMC consists of chopped strand E-glass blended with resin and fillers supplied in continuous rolls. It is manufactured on a continuous belt. A lower cover film is coated with the liquid resin/filler blend, glass rovings are then chopped to lengths of 20–75 mm, and allowed to settle on the moving belt; a second top cover film, also resin coated, is positioned on top of the first and the whole sandwich passed through a set of kneading rolls that homogenize the mixture. On exit from the machine the strip is coiled into rolls of convenient size and stored in the maturation room. Before maturation the resin would be liquid and the compound soft and sticky. After maturation the resin is pasty and nonsticky. The material is typically 1–2 m wide and upward of 5 mm thick.

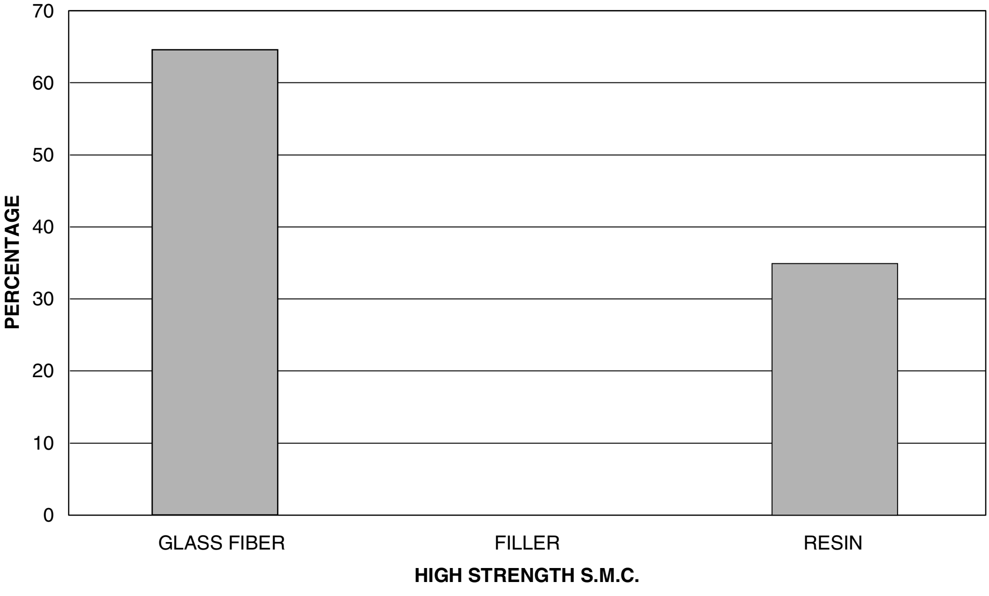
There are a number of variants of the basic SMC, also called SMC-R, for *random*. A standard formulation is 35 wt % E-glass, 40 wt % filler and 25 wt % resin (Fig. 40*a*): This allows a very good surface finish to be achieved but lacks stiffness and strength due to the low glass content (35 wt % $\approx$ 18 vol % $-$ $V_f \approx 0.18$). Higher mechanical properties are realized by using 65 wt % glass and no filler (Fig. 40*b*), but the surface finish is inferior. The fillers are mostly fine chalk powders, often wollastonite, and other processing aids such as soaps, lubricants, and *low profile additives* are also included. The low profile additives are waxes or low-melting-point thermoplastics that migrate to the surface during cure and improve the surface finish. Instead of chopped strand, continuous strips of CRM may be fed into the SMC machine, again with the choice of high glass/no filler, or low glass/high filler. The CRM conveys some molding and property characteristics, which will be discussed further. Another option is to manufacture the compound from woven fabric. The final alternative is to make the SMC by winding continuous rovings and apply the resin onto a large-diameter drum, giving a near uniaxial lay-up (e.g., $\pm 5°$). This is a batch process. When the required thickness is achieved, a top-cover film is applied and the sheet cut across to remove it from the drum. Sheets of up to 10 m long by 2 m wide may be produced this way. The material was originally developed for the manufacture of vehicle leaf springs, where a near uniaxial lay-up and high V_f is required. For all SMC materials the most common resin systems are the unsaturated polyesters and vinyl-esters. Only these materials allow for the convenient maturation thickening process. Vinyl-ester resins give somewhat better performance and are considered premium materials. Epoxy resins are rarely used in SMCs.

12.3 Molding SMC

All the SMC materials discussed are compression molded in heated metal tooling, typically at 120–180°C. The main differences result in the differing flow potential of the different formulations. SMCs made with chopped strand flow extensively when molded, more so when the fiber loading is lower and the glass chopped into shorter lengths. It is normal practice to promote flow by loading pieces of SMC onto the open tool so as to cover only about 50% of the projected area of the mold. There is no attempt to tailor the pieces to the shape of the part, but an optimum pattern must be determined and the charge accurately weighed, to ensure efficient and reproducible mold filling. When the mold is closed, initially under light pressure, the resin in the charge softens and as the pressure is increased the charge flows to fill the mold cavity (Fig. 41). Quite detailed features such as ribs and bosses may be formed, but there is some danger that the resin will bleed through the reinforcement, so that the remote details of the molding may contain insufficient reinforcement (Fig. 42). This tendency is controlled by varying the fill pattern and modifying the pressure cycle.



(a)



(b)

Fig. 40 Two alternative SMC formulations are illustrated in the two bar charts. (*a*) Standard SMC incorporating 35 wt % of random chopped E-glass fiber bundles, with 40 wt % of filler (chalk) and 25 wt % resin. This gives good surface finish but only moderate stiffness. (*b*) This alternative formulation contains 65 wt % of chopped glass with 35 wt % of resin. It molds less readily to complex shapes and has an inferior surface finish, but much greater stiffness and strength. It is widely used for *hidden* structural parts.

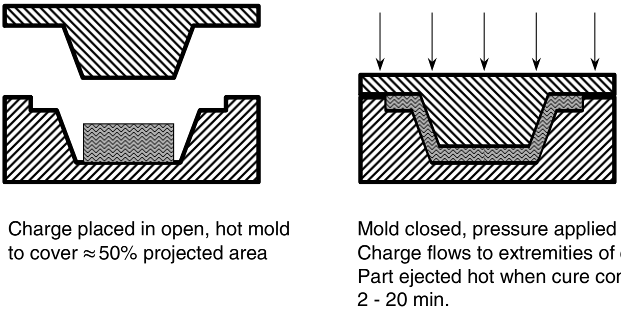


Fig. 41 When molding SMC the charge is placed so that it covers about 50% of the projected area of the mold face. As the mold is closed, under pressure, the charge is heated, the resin softens, and is forced to flow to fill the mold cavity.

Materials with higher fiber content and those manufactured with continuous random mat will not flow as extensively as the standard SMC. It is necessary to tailor the charge more carefully to ensure good molding performance. It is especially important, when using the high fiber content materials that adequate fiber content is realized in all parts of the molding, as these materials are chosen on account of their mechanical performance. An advantage of the CRM reinforcement is that the reinforcement will not *tear* during flow, leaving fiber-denuded zones. This can be a problem with discontinuous strand materials. SMCs made with woven materials and the uniaxial material described above cannot flow to any significant extent in-plane; they must, therefore, be carefully tailored to the shape of the part, and features such as molded ribs are not a possibility. One compromise is to charge the mold with a mixture of woven and random feedstock, e.g., to stiffen a panel while still being able to mold some out-of-plane features. These are highly cost-effective materials and much ingenuity is exercised to exploit their engineering potential.

Once the charge is heated and full pressure applied to the press, curing occurs quite rapidly. According to the size, wall thickness, and complexity of the mold-

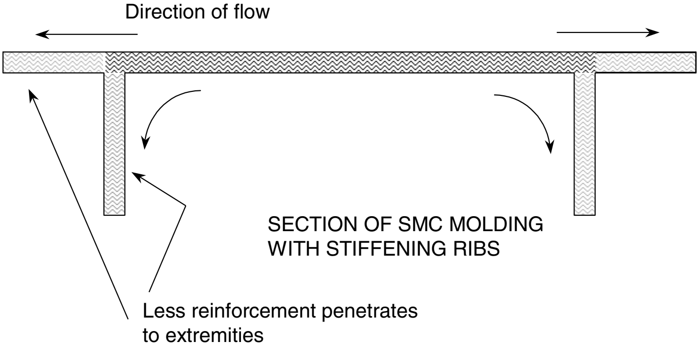


Fig. 42 When forming parts incorporating features such as blade stiffeners, there is a tendency for less reinforcement to flow into these extremities. This can result in weak regions. The problem is countered by careful placement of the charge and by control of the molding parameters—especially the mold closing speed.

ing, cycle times as low as 2 min may be achieved. A cycle time of 10–20 min is more common so that 15,000–30,000 parts/year may be produced from a single press working on a 24-h, 3-shift day. The parts are ejected from the mold while hot. The tools may therefore be operated at constant temperature throughout a molding campaign. This is compatible with the needs of the automobile industry, although much slower that metal pressing. However, assembly and finishing costs are likely to be much less when SMC is used.

12.4 Bulk Molding Compound

This is a similar formulation to SMC except that the compound is normally prepared in a ribbon mixer. The reinforcement is always chopped strand, usually rather shorter than that used in SMC, e.g., 5–20 mm. Standard formulations are typically ≈35 wt % glass, ≈35 wt % filler and the remainder resin. Fillers and processing aids are similar to those used with SMC. The material is extruded as a rough cylinder, matured, and then packed in bags or bulk containers to await final processing.

For compression molding the procedure is similar to SMC. A weighed piece of BMC is placed in the open mold and the molding cycle initiated. BMC is generally used for smaller parts than SMC, so faster processing is the norm.

BMC may also be processed by injection molding, which is becoming the dominant process. Specially developed machines are used, which resemble those used for thermoplastics in some respects. The basis of the machine is a screw pump, which both rotates and reciprocates. The charge is fed into the feed end of the screw by a mechanical *stuffer,* as the BMC cannot be gravity fed. The charge is transported forward by the screw through the barrel, which is heated to a temperature of usually 60–100°C, by means of a water or steam jacket. This is sufficient to soften the resin in the BMC but *not* to initiate cure. A reservoir of plasticized material is built up in front of the screw, which retracts under controlled backpressure to accommodate the forward flow of BMC. The screw is then driven forward to inject the accumulated charge into the hot mold. Pressure is maintained while the charge cures, typically 1–5 min. The rate-limiting step is usually the time needed to heat the charge from barrel temperature to cure temperature and for the cure to be completed. This may be speeded up by using a very fast injection rate. This heats the charge by the work of shear induced in the charge and eliminates the heat-up time. Injection molding of BMC requires high pressures and a very robust molding machine and tooling, which are more costly than the thermoplastics equivalents. Production can be completely automatic and very high production rates achieved. Fine detail can be incorporated into the designs and the technique is widely utilized in the automotive and domestic appliance industries, where BMC parts often replace parts traditionally fabricated from metal castings.

13 GLASS MAT THERMOPLASTICS

13.1 Principles

Thermoplastics are always solids at room temperature, therefore, processing methods must be adapted to accommodate the necessary melt–freeze cycle. Glass mat thermoplastics (GMTs) are the thermoplastics equivalent of SMC.

They are preimpregnated materials in sheet form. The processing cycle consists of heating the feedstock to above the melting point of the thermoplastic matrix and then stamping or pressing the hot material between cold tools. The hot compound flows to fill the mold cavity and then cools rapidly in contact with the cold tool faces, and the melt solidifies. Process cycles can be as short as 30 s.

13.2 Reinforcements and Matrices

In principle any sheet form reinforcement may be used. The terminology GMT implies glass fiber mat reinforcement, and this is the standard commercial product, but it is possible to use both aramid and carbon fibers, either alone or hybridized with glass. Likewise formats other than mat, such as uniaxial (i.e., similar to prepreg) or woven, may be used. The standard commercial materials are mostly based on the use of continuous random E-glass mat, CRM, although some use chopped strand. This choice is driven by economics and optimization of the flow process when the material is stamped. The matrices in commercial materials are either polypropylene (PP) or a polyamide (PA), usually PA 6, 11, or 12. Other thermoplastics have been used and uniaxial and woven materials using PEEK (poly-ether-ether-ketone) and other aromatic thermoplastics are manufactured for high-performance applications. They are discussed in the following section. The choice of PP and PA is based on their relatively low melt temperatures, their melt rheology, and performance. Molten thermoplastics are very viscous and to ensure adequate flow, the fiber fraction and filler loadings are lower than for typical SMCs. This inevitably means that the mechanical properties, especially stiffness, will also be lower. This is compensated by greater toughness, better environmental performance under some conditions, faster processing, and potential recyclability.

13.3 Processing

The feedstock is most often supplied a sheets, 0.5–2 m wide, 1–2 m long, and typically 5–10 mm thick. They have been manufactured by rolling a stack of alternate layers of glass mat and polymer film through heated rolls. The glass mat is not usually completely impregnated. Blanks of a suitable size are cut from the sheets. It is not common practice to tailor the blanks to the molding, but simply to cut a set of blanks, ensuring that the total weight of the charge is correct.

The cut blanks are then heated, usually in a belt feed, tunnel oven, heated by circulating hot air, or by radiant heaters. The heating process must be carefully controlled because the material has low thermal conductivity and too fierce an application of heat could lead to local degradation at hot spots. When the blanks emerge from the oven, the matrix is completely melted. There is often considerable exfoliation, and the 5-mm-thick blanks swell up to 20 mm thick, or more. Although the matrix is molten, the blanks may still be handled as the glass mat holds everything together. This is one reason why CRM is preferred. The hot blanks are then placed onto the bottom surface of the molding tool in a predetermined pattern (cf. SMC). In this condition the exfoliated blanks do not chill excessively. The press is then closed and the charge is squeezed between the tool faces, forcing the material to consolidate and flow to the extremities of the mold cavity. Quite high degrees of draw are possible. Once consolidated the charge is rapidly chilled by contact with the cold tooling, and the tool may be opened and the part ejected. The process is illustrated schematically in Fig. 43. The speed of mold closure is critical. It must be sufficiently fast that flow to the tool extremities occurs before the charge is chilled below the freezing point of the matrix, hence the use of the term *stamping* rather than *pressing*. The speed of the process renders it particularly attractive for mass production industries. Fine detail may be incorporated into the moldings, so that a single GMT part can replace an assembly of pressed metal. In general the surface finish is not as good as low-profile SMC and inadequate where a ‘‘Class A’’ finish is required, e.g., external panels for automobiles.

14 HIGH-PERFORMANCE THERMOPLASTIC MATRIX COMPOSITES

14.1 General

The development of the high-performance aromatic thermoplastics such as PEEK PES (poly-ether-sulfone), and PPS (poly-phenylene-sulfide) provides materials with mechanical and elevated temperature properties that can compete with epoxy resins for composites matrices. There are potential processing advantages, and it has been demonstrated that carbon fiber combined with these matrices provides composites with superior toughness and resistance to some environmental hazards. There has, therefore, been a period of intensive development of these high-performance thermoplastic based materials for aerospace and similar applications.

14.2 Feedstock

In conformity with aerospace practice, which is built around use of prepreg, the thermoplastic systems are produced as prepreg-like impregnated tape, as fine

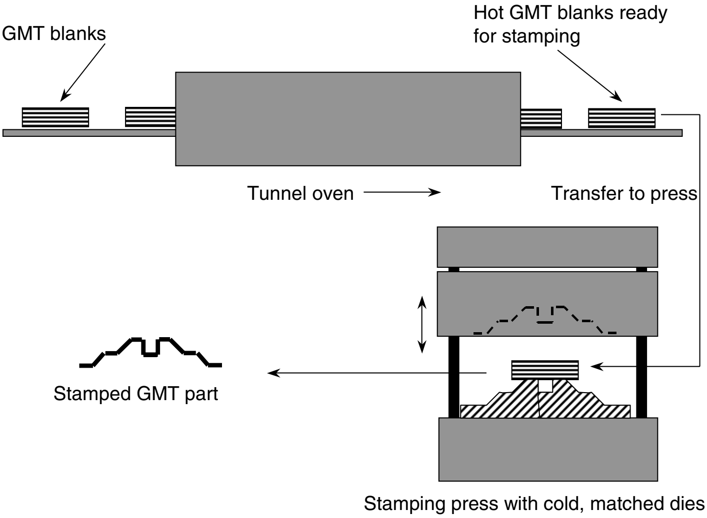


Fig. 43 Glass mat thermoplastic (GMT) forming process is illustrated. Cut blanks of thermoplastic impregnated glass mat are heated by passing through a tunnel oven. The hot blanks are then stamped between hot dies. This forms the part and chills the material making for a fast process cycle.

woven impregnated tape, and as impregnated tow. The normal reinforcement is carbon fiber as it is not generally cost effective to use lower performance fibers with such expensive matrices. The only exceptions would be where specific physical properties, such as radar transparency, were required.

The tape materials are produced in thicknesses comparable with that of conventional prepreg, e.g., 0.125–0.25 mm, much thinner than GMT. Impregnated tow is typically a single tow of 6000 or 10,000 carbon fibers. The sheets are stiff and *boardlike* at room temperature and cannot be draped at all without heating. The tows may be coiled.

14.3 Processing Principles

The principles for processing high-performance thermoplastic composites are similar to those for GMT. The material must be heated to a temperature that melts the matrix; it can then be shaped and must be chilled in the formed shape to freeze the matrix. There are, however, a number of additional considerations.

First, the continuous fiber, high V_f, feedstock material will not flow to any extent when molded. Its drape possibilities are similar to those of prepreg, except that the thermoplastic form must be heated before it can be draped at all. A second consideration is that the melting points of some of the matrices approach 400°C, so that any tooling that needs to be heated to the melt temperature must be steel. Other materials can be used where the hot feedstock is stamped between cold dies. A third point is that some of the thermoplastics are semicrystalline, e.g., PEEK, and their properties are strongly sensitive to the degree of crystallinity. This means that the cooling rate from the melt temperature to below the crystallization temperature must be controlled. Finally, when using high-performance materials, very close control of V_f, fiber architecture, and dimensions is required so that optimum performance is achieved. These factors combine to make processing a more complex operation. The simple preheat and cold stamp process used for GMT is sometimes adequate but, in general, these prepreg-like feedstocks require a more extended period for consolidation while hot.

14.4 Autoclave and Press Processing

Panels of moderate single curvature and low complexity may be molded using either an autoclave or a press. The thin sheets of feedstock are cut to the required profile and stacked in sequence, as for prepreg. The difference is that the material has no tack to retain each ply in position. This may be accomplished by thermal tacking. A soldering iron or similar implement is used to *spot weld* the sheets together as they are laid. When the laminate has been completed, it is placed on the tool and covered with a vacuum membrane. Owing to the high processing temperature, this may need to be a high-temperature stable polymer or elastomer, or even aluminum foil. The assembly is then subjected to vacuum and placed in the autoclave. The temperature and pressure may then be raised so that the matrix melts, typically in the range 250–400°C, and the pressure consolidates the laminate. This process can take longer than for thermoset systems due to the higher viscosity of the molten thermoplastic. Once consolidation has been completed, the whole assembly must be cooled, under pressure, until the matrix has solidified. Furthermore the cooling rate must be controlled through the critical crystallization region. This may entail incorporation of water cooling in the tooling. The autoclave may then be opened and the parts demolded. This is clearly a rather time-consuming procedure.

An alternative method is to use a press tool with means of heating and cooling. The tooling may be either single-sided, working against an elastomer counter tool, or double-sided matched tooling. The choice depends on part complexity and process temperature. The lay-up procedure is similar to that described above, the laminate is then placed into the tooling, which is heated, and pressure is applied in a controlled manner. The full pressure and temperature are held while consolidation is effected and then cooled down under pressure.

In favorable cases the stacked laminate may be preheated in an oven, the hot laminated transferred to hot tooling, pressed, and consolidated. The press is then opened and the hot part transferred to another set of cold tooling in a separate press, where pressure is applied while the part cools. Auxiliary tooling can be used to facilitate the transfer from the hot to the cold press tools. This method is much faster as it is not necessary to heat and cool the molds during each cycle. It is also more thermally efficient. The disadvantage is that two sets of tools are required, although the cold tools do not need to be made from high-temperature stable materials.

14.5 Diaphragm Forming

Diaphragm molding is a variation of the vacuum forming process used for simple thermoplastics. The stacked laminate is held between a pair of deformable diaphragms, and then, after heating, formed into a single-sided tool by vacuum, gas pressure, or a combination of these. The diaphragm materials need to be stable at the processing temperature but the shaped tool remains cool. Diaphragms may be high-temperature stable polymer or elastomer films, suitable for short service at temperatures of up to about 300°C or superplastic aluminum alloy for higher temperatures. The process is reasonably fast but the cost of the disposable diaphragms is a significant added cost.

14.6 Economic and Performance Implications

There has been much speculation, over the past 20 years, that high-performance thermoplastics matrix composites could challenge the dominant position held by carbon/epoxy thermoset systems. This has not happened! The initial prospect of fast, cheap processing proved to be something of a myth and material costs have remained high. The ease of processing of the low V_f GMT systems does not apply with high V_f, continuous uniaxial fiber laminates, which are probably even more difficult to process than the thermosets. There are applications where thermoplastics systems are more cost effective, mainly for less complex shapes, and it is undisputed that the thermoplastic matrix composites offer a combination of stiffness and toughness that cannot easily be matched with thermoset systems. The two classes now operate side by side with thermosets the senior partner.

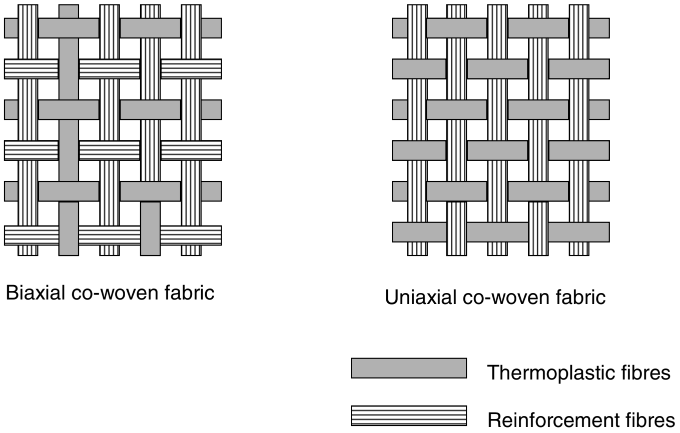
15 COMMINGLED THERMOPLASTIC MATRIX COMPOSITES

15.1 Principle

Several strategies have been developed to deliver thermoplastic matrix composites feedstock in a more drapable format than the prepreg-like materials discussed in the previous section. This is accomplished by the combination of the reinforcement fibers with the matrix in a thin foil, tape, fiber, or powdered form. These materials may be supplied as mingled tows or in sheet formats. When the material is subjected to heat and pressure, the thermoplastic component melts and is infiltrated into the reinforcement by the action of the pressure. The key to the viability of these materials is that the infiltration distances are very short, due to the finely aggregated state of the feedstock. There are three main types of product in this category, co-woven fabrics, co-mingled (also known as *commingled*) tows, which may in turn be woven into fabrics, and fiber/powder combinations in tow form, again with the possibility of being woven.

15.2 Co-woven Fabrics

These are simply fabrics woven from a combination of tows of reinforcement fibers and tows of thermoplastics fibers. A prerequisite is that the matrix material is available or can be drawn into a suitable fibrous form. This is no problem with a range of commodity thermoplastics that are used as textiles. These include polypropylene, several types of polyamide (nylon), and polyesters. Some of the higher performance thermoplastics, e.g., PEEK, may also be drawn into fibers, but as there is no established mass market for textiles in these materials costs are likely to be much higher. It is also possible, but not extensively practiced, to use ribbons of thermoplastics tape rather than tows for the thermoplastic component. The two materials are combined in proportions corresponding to the required final V_f. The most usual arrangement is simply to alternate the tows of both warp and weft (fill) between the two materials, shown in Fig. 44. The linear densities of the tows are chosen to give the correct proportions of reinforcement to matrix. The resulting product is a highly drapable fabric. It may be handled in the same manner as any other reinforcement fabric. These materials have no natural tack but may be retained in position during lamination by *spot welding* using a soldering iron or similar device or by use of spots of a compatible


Biaxial co-woven fabric

Uniaxial co-woven fabric

Fig. 44 Co-woven fabrics are woven from a combination of tows of reinforcement (e.g., E-glass or carbon fiber) and tows of thermoplastic fibers (e.g., polypropylene or polyamide). Various uniaxial and biaxial reinforcement geometries are possible. On hot pressing the thermoplastic component melts and flows to form the matrix.

adhesive. Once assembled, the laminate is consolidated by heat and pressure. This may be carried out in an autoclave with vacuum bag, in which case the mold/laminate assembly must be cooled to freeze the matrix before demolding. The alternative is to heat the laminate separately, e.g., in an oven, and then press between cold tools. The problem is to ensure that full infiltration is affected. The problem with co-woven fabric is that the amount by which it must be consolidated is greater than with conventional preimpregnated materials, because the interstices between the fibers, both reinforcement and matrix fibers, and between the woven tows are open, i.e., filled with air. This space, typically 30%, must be eliminated to achieve a pore-free laminate. Another factor is that the mean infiltration distance is of the order of one tow width, e.g., ≈ 1 mm, while this is quite short, a significant time interval is necessary for the very viscous liquid thermoplastic to fully infiltrate the tow. For this reason there has been intensive development of the co-mingled fiber systems discussed below.

15.3 Commingled Tow Materials

In these materials reinforcement fibers are combined with thermoplastics filaments within a single tow (Fig. 45). The result is a textilelike tow that may be woven or placed directly. The advantage of this system is that infiltration distances are only of the order of the filament diameter—a few microns. The most successful commercial system is a combination of E-glass and polypropylene fibers, marketed under the name Twintex by the French company Vetrotex. This material is available in a variety of woven fabrics that are easily draped and in favorable circumstances may be processed by preheating the preform and pressing between cold dies. More complex shapes and thicker sections may require hot pressing followed by transfer to cold tools for chilling. It is also possible to filament wind or to use tow placement techniques where the tow and the workpiece are heated locally at the point of application of the tow, so that it is

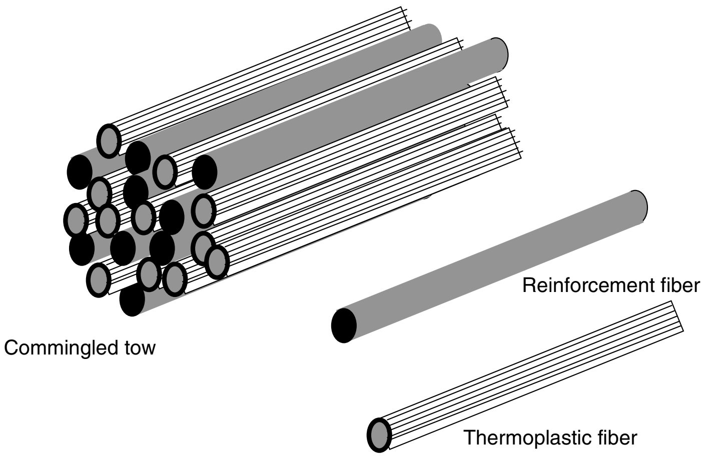


Fig. 45 Commingled tows are formed from an intimate, one-to-one, combination of reinforcing fibers and textile fibers. These materials are now available commercially. They may be woven into fabrics or used directly in tow placement technologies. Their main advantage over co-woven materials is that the flow path is much shorter and processing therefore much faster.

progressively welded into position. This technique holds great promise for the future but has yet to be perfected as a viable commercial process. The commingled tows may also be used as feedstock for pultruding. Again, this is a developing technology of considerable promise but is currently not competitive with thermoset-based pultruding.

The commingling principle is also being developed using other thermoplastics as the matrix. There would appear to be considerable potential in the use of polyamide fibers, especially PA11 or PA12, which have melting points of about 200°C and low melt viscosity. These matrices in combination with carbon fibers offer better mechanical properties and elevated temperature performance than the E-glass/polypropylene materials at a cost that is attractive for automotive and similar applications. There has also been development using high-performance aromatic thermoplastics such as PEEK with carbon reinforcement. Such materials are intrinsically much more expensive and need to be processed at temperatures of about 400°C and are only of interest where high performance is essential. However, the development of the commercial commingled systems means that an installed plant could be used for processing the more exotic combinations in the future. Overall this is a technology with great potential.

15.4 Fiber–Powder Combinations

Another technique, which has enjoyed some commercial success, is to combine reinforcement fibers with thermoplastic polymer powders in a towlike material. The reinforcing fibers are passed through a fluidized bed or a slurry of suspended polymer powder. The fiber bundles are *teased* open, using gas or liquid jets so that powder is entrapped between the fibers. The whole bundle is then encapsulated in a thin-walled tube of the polymer formed by wrapping a tape of polymer foil around the bundle. The tape is then welded using a hot gas jet or an ultrasonic welding device. The resultant product is a flexible tube containing the reinforcement fibers and the entrapped powder particles. On heating and pressing, both the foil tube and the powder particles are melted and flow to infiltrate the fibers and form the matrix of the composite. This material may also be woven, although not as conveniently as the commingled fibers, and may also be used as tow placement, filament winding, and pultrusion feedstock.

16 SRIM OF MONOMER PRECURSOR THERMOPLASTICS

In general, one of the key attractions of thermoplastics composites is that there are no chemical reactions during the final processing stages. There is, however, a small group of materials that may be processed by *in situ* polymerization of monomeric precursors to produce thermoplastic matrices. These are mainly the polyamides, which are polymerized by *ring-opening* chemistry. These are notably PA11 and PA12. The monomers, also known as *lactams,* are liquids of very low, waterlike, viscosity. They are quite unstable, and addition of small proportions of an initiator or catalyst will induce a rapid molecular ring opening followed by addition polymerization, forming the thermoplastic. The reactions are very fast and may be completed in periods of less than one minute. The process is, thus, eminently suitable for use in a SRIM-type process, where dry preforms of the reinforcement are placed in the mold, and the liquid precursor is then injected. The equipment is basically similar to that used for conventional

RIM or SRIM, but a mixing head of different design is needed as only a small proportion of initiator/catalyst must be mixed with the monomer. This is the critical operation. Provision must also be made to prevent overheating due to the reaction exotherm. The very low viscosity of the monomer ensures rapid and complete infiltration, and the rapid polymerization allows fast cycling. This is a very specialized technology applicable only to this one class of polymer, at least at the present time. Carbon fibers are particularly well suited for this process as a strong interface bond is formed with the polyamide. This combination provides considerable potential for mass production of higher performance components.

17 INJECTION-MOLDED SHORT-FIBER-REINFORCED THERMOPLASTICS

17.1 General

Short-fiber-reinforced thermoplastics (SFRTPs), are often classified as *reinforced plastics* rather than *composites,* but since they are one of the most important groups of commodity materials, it is useful to include them in the present discussion. The total quantity of SFRTPs manufactured each year is comparable with that of the basic E-glass/unsaturated polyester GRP materials. Together they account for more than 80% of the composites market. They are injection-moldable materials consisting of thermoplastics matrices with discontinuous fibers, normally in the length range of 10 mm down to <0.1 mm. The fibers are much shorter than those in the other composites systems discussed in the previous sections. The fiber loading is also lower than in most other composites, rarely more than $V_f \approx 0.2$, although the fiber content is often expressed as a weight percentage. Thus, a polyamide with 30 wt % of E-glass would have an equivalent V_f of about 0.18. The very short fibers and low fiber loading are necessary to allow the compounds to be easily injection molded and to achieve a good surface finish. Somewhat better mechanical properties may be realized at the expense of surface finish if longer fibers are used at higher loadings. SFRTPs are often formulated with particulate fillers, e.g., talc or chalk, and sometimes rubber additives as well as the fibers to obtain the optimum balance of properties and cost.

17.2 Principal Systems

Fiber-reinforced grades of most of the commodity and engineering thermoplastics are offered by the polymer suppliers and compounders. The most important commercial systems are those based on polypropylene, the polyamides, the acetal resins (Delrin), and the thermoplastic polyester family. In all of these materials the principal fiber used is E-glass. Glass-reinforced grades of the higher performance thermoplastics such as polyphenylene sulfide and PEEK are also offered, and carbon fiber is also used with polyamides and the higher performance materials.

17.3 Compounding

Short-fiber-reinforced thermoplastics are invariably precompounded. There are two principal methods. In the first either continuous fiber rovings or rovings precut into short lengths (e.g., 10 mm) are fed into a screw extruder through which the plasticized polymer is also transported. The fiber and polymer are vigorously mixed as the blend passes through the screw and are finally extruded through a *spaghetti* die, and the extrudate cut into short pellets. The fibers are broken into short lengths during this process. The output is cylindrical granules, typically 4 mm diameter and length. The granules contain a semirandom array of very short fibers, typically less than 1 mm long. These granules are used as feedstock for the injection-molding process.

In the alternative process, continuous fiber rovings are impregnated with the polymer, either in a specially adapted extruder with a cross-head die or by a variant of the pultrusion process. The output is a continuous rod of about 4 mm diameter. These rods are then chopped to lengths of about 10 mm, and this material used as feedstock for the injection molders. The distinction between the two compounding processes is that, in the former, the granules contain very short fibers, while in the latter the fibers are the full length of the pellets. Further fiber breakage occurs during injection molding, but the moldings made with the long-fiber granules will contain fibers of much greater average length. This induces superior stiffness, strength, and toughness, but surface finish may be poorer. A schematic of the structure of both types of molding pellet are shown in Fig. 46.

17.4 Injection Molding

Modern injection-molding machines are built around screw plasticizers that can be rotated and reciprocated. The charge of compounded granules is gravity fed from a conical hopper into the feed section of the screw. From here the charge is carried forward by the action of the rotating screw. The charge passes down the heated barrel, where it is melted and subjected to a strong mixing action, which homogenizes the melt. The plasticized melt builds up at the end of the

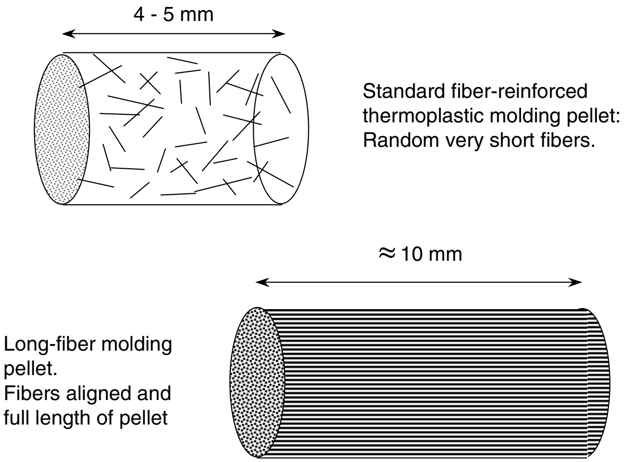


Fig. 46 Illustration of two types of injection-molding pellet for short-fiber-reinforced thermoplastics. The standard pellet is formed by extruding a compounded mixture of polymer and fiber. It contains very short fibers in a random orientation distribution. The long-fiber pellet is made by a cross-head extruder or a variation on the pultrusion principle. The fibers are oriented along the full length of the pellet that are typically cut into 10 mm lengths.

screw, against a closed discharge valve. The screw is forced back in the barrel by the pressure built up against the valve. When sufficient charge has been accumulated, the screw rotation is stopped, the valve is opened, and the screw thrust forward by a hydraulic ram, which injects the charge into the mold. The mold is relatively cool and the pressure is maintained (the *dwell*) until the material in the gate has frozen. At this point screw rotation is restarted and the next charge prepared. Meanwhile when the material in the mold has cooled sufficiently, the mold may be opened and the part ejected. The whole cycle is completed in times ranging from as little as 10 s to a few minutes. The critical rate determining factor is usually the length of time required for the charge to cool in the mold prior to ejection. This will be longer for larger moldings and thicker sections where heat transfer through the mold is less efficient. A schematic of the essential parts of an injection-molding machine is shown in Fig. 47.

The operation of the machine must be carefully controlled to assure that the specified mechanical properties are obtained, that the surface finish is acceptable, and that the optimum production rate is achieved. The main controls are the barrel and mold temperatures, the screw backpressure, the injection pressure and rate, and the dwell time. A higher barrel temperature will increase the maximum plasticization rate, usually expressed in kilograms/hour. Too high a temperature may lead to degradation of the polymer. A lower mold temperature will speed up the cooling cycle but often leads to poor surface finish. Better surface finish usually entails a compromise, and mold temperatures are typically held at 40–100°C with polypropylene and polyamides. This increases cooling time and, hence, the total cycle time. The screw backpressure regulates the pressure in the melt reservoir prior to injection. A higher backpressure gives more thorough mixing but also breaks the fibers into shorter lengths. This compromises mechanical properties but tends to improve surface finish. Faster injection rates result in better mold filling due to the shear thinning (pseudoplastic) behavior

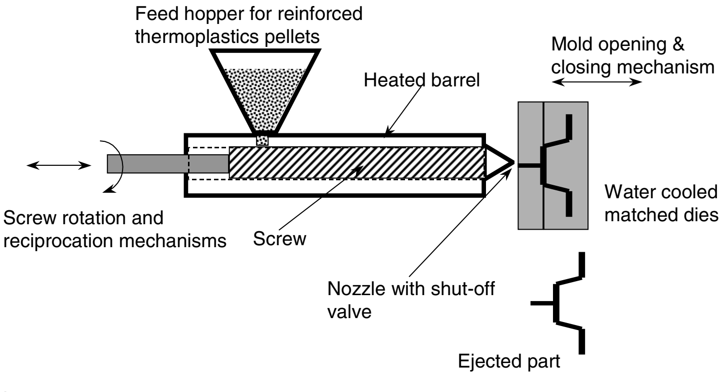


Fig. 47 Schematic of an injection-molding machine for reinforced thermoplastics. The pellets are charged into the hopper, from which they are gravity fed into the entry section of a screw pump. The charge is carried forward and melted in the heated barrel, while the screw retracts under the pressure of the melted charge at its tip. When sufficient charge has accumulated, the screw is forced forward and the nozzle valve opened allowing the molten charge to be injected into the cold mold.

of polymer melts, where apparent viscosity decreases with increased shear rate. There is also adiabatic heating, where the injected charge is heated through the shear work performed during injection. Overheating can lead to *burning* and degradation. Finally, higher pressure maintained during the dwell period tends to compensate for the shrinkage that occurs as the polymer freezes. In this way a sharper impression of the mold and better control of dimensions is ensured.

When molding SFRTPs, the principal concerns are to control the fiber length distribution and the fiber orientation distribution to ensure that optimum performance is obtained. The fiber length distribution is affected mainly by the choice of feedstock, long or short fiber, and the backpressure and injection rate settings. The orientation distribution is flow dominated. Convergent flow leads to fiber orientation in the flow direction, while divergent flow has the opposite effect. The flow pattern is mainly determined by the geometry of the part and the placing of the injection point(s) or gate(s). Comprehensive suites of software (e.g., Moldflo) are available that allow the flow in the mold to be visualized so that optimum gate placement may be determined at the tool design stage.

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CHAPTER 35

ADVANCED CERAMICS PROCESSING

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1 OVERVIEW OF CERAMIC PROCESSING

Processing of ceramics is carried out through the consolidation of loose powders to form polycrystalline objects. The majority of ceramics are sintered, using solid-state sintering or liquid-phase sintering [See, e.g., Rahaman (1995), Barsoum (1997)]. Generally, sintering occurs at temperatures below the melting point for single-phase ceramics or below their liquidus for multiphase ceramics, although a liquid phase may be present temporarily during sintering. The distinction that sets the majority of ceramics apart from the minority is the fact that the majority is sintered below the melting temperature by solid-state transport. The minority are those processed above the melting temperature, in particular, glasses and glass-ceramics. Therefore, the key to successful processing of ceramics is finding energy sources that can drive the transport processes.

In most cases, the source of energy is surface energy. The elimination of surface energy, where surfaces of loose powders are converted to grain boundaries between particles, is what accounts for most of the energy available for sintering. There are countless examples of ceramic products where the conversion of this energy has resulted in a successful outcome. Nevertheless, there can always be improvements in ceramic processing. Therefore, the focus of this chapter is on new sources of energy to drive the consolidation and better, more efficient uses of this energy to reach the goal of a well-consolidated, polycrystalline ceramic.

Handbook of Materials Selection. Edited by Myer Kutz

1.1 Microstructure

When it comes to describing the outcome of processing in ceramics, the focus of that description is the microstructure (Allen and Thomas, 1999). To put this in perspective, the terms we use in describing microstructure need to be defined. As such, microstructure is described in terms of grains, grain boundaries, and porosity. For single-phase material, this is straightforward, but far more complicated for multiphase materials. Because the starting point for processing is loose powders or particles, the first feature in the microstructure is described as necks, which are the contacts between particles. Next, we distinguish between a single crystal, which is a "perfect" crystal without interruption, and a grain, which is a single crystal of finite size. Once there is consolidation within a powder compact, what was once called a particle becomes a grain. The regions of disorder between these grains become grain boundaries. In the grain boundaries, we often find a second phase, which is the remains of a liquid phase.

Another way of looking at the microstructure is focusing on the porosity, which is the collection of voids. Initially, the porosity is interconnected. Gradually, material transport fills in the voids, bringing more area of grains into contact. Triple-grain junctions are the typical location of closed porosity. In the end, the polycrystalline solid takes on its final microstructure through grain growth and coarsening. In short, the goal of sintering is to turn the loose powders into a well-consolidated polycrystalline solid.

With these terms, the features of the microstructure can be quantified. Quantities such as pore volume, percent theoretical density, and grain size can be used to draw the correlations we are familiar with in materials science. No materials science student can graduate without learning the structure–processing–properties–performance paradigm known as SP^3. The structure of the material determines the properties, and the structure, in turn, is determined by processing.

1.2 Motivation for Improvements

An alternative way to view ceramic processing is in terms of industrials needs. Suppose there is a need, that can be satisfied by a ceramic product. The engineering factors involved are shown in Fig. 1. The role of processing weighs more heavily on the manufacturing side. Because issues of cost and reproducibility balance the function of the product, the eventual implementation of the ceramic product may hinge on whether or not the product can be made in a practical way.

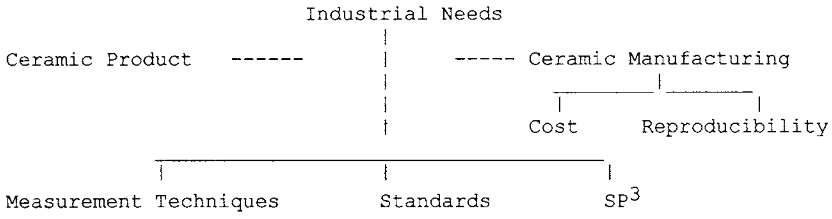

Fig. 1 Industrial issues relevant to ceramics.

1.3 Manufacturing versus Processing

Is ceramic manufacturing comparable in scale to metals fabrication or comparable in automation to pharmaceuticals production? The easy answer would be no. What efforts have been made to introduce continuous production, automation, and continuous improvement into ceramic manufacturing? How much intelligent processing is used in ceramic manufacture? By intelligent processing, we mean that we can insert diagnostics in the ceramic process that allow us to evaluate physical properties during the process. The goal of intelligent manufacturing is achieved when we can correct the process to stay within acceptable values of our physical properties before we reach the final product.

These questions about intelligent manufacturing are important to the health of ceramic manufacture in a setting where at times more than one material or design satisfy the industrial needs. Clearly, there are some ceramic products that cannot be replaced by other material systems, but there are other ceramic products, such as glass containers, that are seriously threatened by other materials, such as plastics. To answer these questions about ceramic manufacture, and, in particular, about manufacture of advanced ceramics, requires a careful look at individual segments of the industry. Moreover, they are useful questions to keep in mind, as new processing technologies are introduced.

In terms of manufacturing a product that meets customer requirements, the technical issues include quantitative specifications of properties, process improvement and "zero defects." The nontechnical issues include government regulation and availability of investment for capital and product development. Overall, the goal of manufacturing is to produce a high-quality product rapidly, in an economical way, and in an environmentally acceptable manner.

1.4 Back to Basics

To improve processing means understanding the fundamental mechanisms dictated by thermodynamics and kinetics. What goes on in ceramic processing is listed in Fig. 2, connecting raw material to finished part. In taking raw material to finished part, how many of these changes can be followed in real time? How many of these changes are predictable, reproducible, and what is the accuracy of the measurement that tells us that they are reproducible? These questions are the real challenges of ceramic processing and ceramic manufacture.

2 CONVENTIONAL PROCESSING

As with any materials process, the unit operations we evaluate are powder preparation, forming, consolidation, and sintering. For each step, it is necessary to

Raw Material → Process-induced Changes → Finished Part

Mass change	(e.g., calcination)
Shape change	(e.g., sintering)
Structure change	(e.g., solid solution)
Phase change	(e.g., polymorphic transformation)
Chemical change	(e.g., oxidation)

Fig. 2 Process-induced changes that link raw material to finished part.

Table 1 Examples of Structural and Functional Ceramics, Principally Single-Phase, Produced by Sintering

Ceramic	Property	Application
Structural		
ZrO_2	High-temperature strength	Furnace lining
Si_3N_4	Wear resistance	Bearings
SiC	Corrosion resistance	Heat exchanger
TiC	Hardness	Cutting tools
Functional		
$BaTiO_3$	Ferroelectricity	Capacitor
Al_2O_3	Electronic insulation	Substrates for electronic packaging/ Spark plugs
$BaO \cdot 6Fe_2O_3$	Hard magnet	Ceramic magnets
$(Zn)Fe_2O_3$	Soft magnet	Transformer core
$LiNbO_3$	Nonlinear optics	Optical switches

understand the mechanisms and the extent to which we can model the steps with thermodynamics or atomistics. A key question at each step is: What needs to be measured and how can it be measured? Does the instrumentation exist that allows us to follow a ceramic process in a continuous way? Are we measuring the parameter directly or indirectly? When a process seems to be going out of control, how can we fix it and can we fix it without going back to the beginning?

The steps in the fabrication of polycrystalline ceramics are divided between those before firing and those during and after firing. Many excellent treatments of ceramic processing have appeared over the last few years. A comprehensive handbook on the subject treats each step in detail (Schneider, 1991). For conventional ceramics (e.g., whitewares), ceramics produced in large volume (e.g., abrasives), and ceramics used at high temperatures (e.g., refractories), there are textbooks that cover the processing and the applications [see, e.g., Reed (1995), West and Hench (1990), Richerson (1992)]. Table 1 lists typical single-phase sintered ceramics and their uses.

Some of the common steps in ceramic manufacture are shown in Fig. 3.

Rather than review what is now well known, this discussion will cover processes that are new. These processes may not be suitable for production of all ceramics in widespread use. Instead, these processes are variations of conventional ways, in some cases, and radically new ways, in other cases, for making ceramics. In all cases, the advantages of these processes over old methods will be highlighted and evaluated.

Raw Material ->	Size ->	Form ->	Dry ->	Fire ->	Finish
Find/Mine	Crush	Press	Binder	Sinter	Machine
Beneficiate	Grind	Extrude	Burn-out		Polish
		Injection Mold			
		Cast			

Fig. 3 Unit operations in manufacturing ceramic part.

3 ADVANCED PROCESSING

When it comes to advanced ceramics, we can assume that there is value added in the product that justifies advanced processing. If there is no value added, then we might as well follow the methods developed thousands of years ago for pottery. It would be foolish to suggest that we are abandoning pottery and whitewares or that we ever will. To the contrary, whitewares constitute a major part of the ceramic industry, and there are continuing improvements in their manufacture, with respect to increasing their environmental friendliness, recycling, performance, and economics.

The need for improving conventional processing has been well served [see, e.g., Lange (1989), Lewis (2000), Sigmund et al. (2000)]. When it comes to conventional powder processing, the needs are water-based systems to replace organic-solvent-based systems, denser suspensions, better powder stabilization schemes, and more *in situ* diagnostics. While addressing these issues is critical to the health of the ceramic industry, the long-term survival and growth of ceramics require some radical new processing schemes. The new technologies are the basis for the remainder of this chapter. The interested reader will find that conventional ceramics are treated thoroughly in many excellent textbooks [see, e.g., Reed (1995)] and there is not enough room in this review for a full treatment.

In looking for what is new in ceramic processing, three directions appear. One can look for new energy sources for processing, such as microwave heating, self-propagating reactions, mechanochemical processes, or reaction bonding. One can look for new ways to control particle characteristics, such as size (e.g., nanoparticles), shape (e.g., equiaxed particles), metastable phases (e.g., rapid solidification), or templating (e.g., seeding). Alternatively, one can look for new building blocks, such as polymer precursors or sol–gel processing.

3.1 New Energy Sources

Microwave Processing

What is microwave processing? From the initial attempts in around 1979 to sinter ferrites with microwaves, it has been know that certain materials show heating effects through dielectric loss when irradiated with microwaves at a frequency of 2.45 GHz. Ordinarily, microwaves, which are located on the electromagnetic spectrum between infrared and radio waves, correspond to energies characteristic of molecular rotation. This explains the usefulness of microwaves in reheating food, which contains water molecules. In addition, selected ceramic compositions show rapid heating through the use of microwaves. While materials with low loss tangents are transparent to microwaves and materials, such as metals, with high loss tangents reflect microwaves, it is the materials with intermediate loss tangents that can benefit from microwave treatment. In the case of ceramics, where an ion jump relaxation mechanism gives rise to polarization in an applied electric field, there is the possibility to use microwaves to assist sintering [see, e.g., Katz (1992), Clark and Sutton (1996)]. The advantages of microwave heating are experienced through volumetric heating, and rapid heating, resulting in increased kinetics. The danger of microwave heating in ceramics is nonuniform susceptibility, leading to localized hotspots, nonuniform grain growth, or agglomeration. Microwave sintering has been investigated in oxide ceramics primarily, as well as nonoxides. The greatest use of microwaves in ceramics processing is in drying, but the use of microwaves in sintering is promising in cases where volumetric heating and small grain size are desirable.

Self-Propagating Synthesis

What is self-propagating high-temperature synthesis, often abbreviated SHS? It is a method for preparing composites that involves a chemical reaction that is exothermic. Once the reaction is initiated, the conversion of chemical energy to thermal energy is used to consolidate a powder compact into a composite. The product of SHS is rarely homogeneous or free of pores, but the kinetics of the consolidation is very rapid.

For example, a composite of Al_2O_3–ZrO_2–Nb can be produced [see, e.g., Holt and Dunmead (1991), Munir (1988), Yi and Moore (1990)]. Initially, reactant powders are milled together and pressed into a simple shape. Beginning with a compact of niobium oxide and an intermetallic such as Al_2Zr, it is possible to heat the compact to around 900°C to ignite the reaction. An ignitable powder in this case might be a mixture of aluminum, iron oxide, boron, and titanium on the surface or in a layer in the compact. Reactions in the igniter layer at the ignition temperature set off a self-propagating wave, which generates an adiabatic temperature as high as 2300°C. This temperature is sufficient to melt a eutectic of Al_2O_3 and ZrO_2. The wave propagation velocity depends on the particle size and the presence or absence of liquid phase. The propagation is rarely steady state, and while it is hard to control, it is reproducible. The resulting composite contains Nb metal dispersed in an Al_2O_3–ZrO_2 matrix. Depending on the size of the starting powders, the ignition can generate stable wave propagation through the compact that influences the matrix composition. The SHS process, as practiced in ceramic matrix composites, has been described as burning a cigar in a bell jar. The approach has been expanded to many technologically important systems, well beyond the original class of thermite reactions. Other ceramic composites produced by SHS include TiC–NiAl and TiB_2–NiAl, as well as, compounds such as $MgCr_2O_4$ and $MgAl_2O_4$.

The advantages of SHS are the rapid nature of the process and the reduced energy consumption. These advantages are balanced by the difficulty experienced in controlling the process and the restriction to systems where exothermic reactions occur. Nevertheless, there are many solid–solid and solid–gas systems that experience exothermic reactions once they are ignited, and further work is being pursued.

Mechanochemical Synthesis

What is mechanochemical synthesis? It is a process that uses mechanical activation to bring about chemical reactions instead of using calcination at moderate to high temperature to accomplish the same result [see, e.g., McCormick and Froes (1998), Gilman and Benjamin (1983)]. For example, to prepare a multicomponent oxide such as lead zirconate titanate (PZT), it is possible to mix powders of PbO, TiO_2, and ZrO_2, beginning with high-purity commercial powders, first in a ball mill in ethanol for 48 h, followed by drying, and finally in a shaker mill at 900 rpm for up to 25 h (Lee et al., 1999). The high-energy milling leads to mechanical activation. A schematic flowchart of the process is shown in Fig. 4. At the same time, the surface area is increased, and the so-called mechanical alloying of the powders yields crystal sizes that give broadened X-ray diffraction peaks. The powders that exist following the milling do not give crystal patterns for the individual components. Rather, the appearance of the X-ray diffraction pattern suggests an X-ray amorphous or metastable form of the PZT phase. When carried out properly, the mechanically activated powder converts to crystalline PZT upon heating without the usual intermediate phases of PZ or PT. In addition, the mechanically activated powder sinters more readily than conventional powders. While the effects of high-energy ball milling are generally favorable, there are some drawbacks associated with handling fine particles, particle aggregation, and lead loss.

Originally, mechanochemical synthesis was designed for alloying metals and intermetallics. Nowadays, many multicomponent ceramics, especially technologically important ferroelectric compositions, are prepared using mechanochemical activation. The benefits are seen as (a) a particle size refinement, (b) partial amorphization, and (c) and creation of distributed nuclei of the desired phase during the mechanical treatment. The "chemical" part of mechanochemical processing is said to be the chemical reactions that yield the nuclei of the equilibrium phase, according to the phase diagram. The solid-state reactions occur during the mechanical activation, rather than during thermal activation. This claim is supported by the observation that the nuclei increase in number and size as the time of the mechanical treatment increases, where thermally activated growth would require much higher temperatures. Similarly, the exotherm observed for the formation of perovskite in the thermal analysis of a conventional powder is not observed for the mechanically activated powder because the perovskite phase already exists. Consequently, the desired phase, typically perovskite, can be developed without significant pyrochlore, where conventional processing may require high pressure and high temperature to develop perovskite.

Many ternary oxide systems have been investigated and the mechanochemical treatment has been found to be beneficial. Where mechanochemical treatment is not recommended is systems that are not reactive, those that show agglomeration rather than size reduction, and those that require some additive to activate the powder where the additive, in turn, becomes a contaminant. Alternatives to me-

Powders -	Mix -->	Dry -->	Activate -->	Sinter
0.5-50 µm	in ethanol	80°C	Shaker Mill	Press/
PbO	Ball Mill	Grind/	900 rpm	Compact
TiO_2	48 h, RT	Sieve (200 mesh)	25 h, RT	1100°C
ZrO_2	ZrO_2 balls		WC balls 20:1 balls/powder	

Fig. 4 Schematic flowchart for mechanochemical processing of PZT.

chanochemical synthesis are, therefore, some of the other methods described, such as sol–gel processing, hydrothermal synthesis, pulsed laser ablation, or metal organic decomposition.

Reaction Bonding

What is reaction bonding? To answer this, a good example is an aluminum-oxide-containing composite, referred to as reaction bonded aluminum oxide (RBAO). This technique begins with a porous alumina preform that is infiltrated with aluminum metal powders, or a compact of aluminum oxide and aluminum metal powders [see, e.g., Wu et al. (1993)]. The aluminum metal is oxidized as the compact is heated up to about 1000°C and the compact densifies on continued heating to about 1400°C and higher. The process is "near-net shape" in that the part changes by sometimes as little as 1% in dimension. The mechanism for the growth of oxide is the diffusion of oxygen through the polycrystalline alumina. The oxidation kinetics can be controlled by additions of other oxides, such as zirconia.

A variation of reaction bonding is the "directed metal oxidation," or DIMOX, process [see, e.g., Nagelberg et al. (1992)]. In this case, there is a growth front presented by the breakdown of the alumina film on fresh aluminum melt. The oxidation proceeds by diffusion of oxygen through the melt until the metal is depleted. Similar to RBAO, the kinetics are controlled by alloy additions, in this case, commonly MgO.

In all cases where reaction bonding is used, including earlier studies on reaction-bonded silicon nitride (RBSN), there have to be favorable thermodynamic conditions for the reactions to occur. The reactions bear some similarity to SHS. The advantage of the process is the near-net shape capability for composites that are difficult to densify by conventional means. The difficulty, however, is that the approach is limited to a few reactive systems. Nevertheless, there are critical applications for these materials, such as lightweight armor, that cannot be satisfied in any other way.

3.2 New Shapes

Nanotechnology

What aspects of nanotechnology are relevant to advanced ceramic processing? For a given application, there are now several suitable methods for preparation of nanostructured materials with controlled chemical and physical characteristics. One should ask, however, what is the critical nanograin size that enhances a material's properties for a target application? These preliminary questions need to be asked before entering the expanding area of nanopowders and nanomaterials.

The motivation for synthesizing nanostructured materials is the discovery of physical and chemical behavior exhibited at this size dimension that is neither described by single molecules nor by conventional materials [see, e.g., Siegel (1991), Gleiter (2000)]. Nanopowders have been prepared using vapor- and solution-based methods. The reason is that these methods can be adjusted to avoid hard aggregation and morphology control problems.

One vapor phase technology is a combustion flame system, based on a flat-flame burner, which is capable of synthesizing nonagglomerated nanopowders. The temperature distribution in the flame precisely defines the zones of nucleation, growth, and aging to enable the formation of monodisperse nanoparticles. Equiaxed ceramic nanopowders with a narrow particle size distribution as small as 10 nm have been prepared (Hahn, 1993, 1997).

One solution-phase technology is the hydrothermal method, where solutions or suspensions are heated under pressure to precisely control the nucleation, growth, and aging of the crystallites. In comparison to other solution-based technologies, hydrothermal methods offer the ability to prepare anhydrous crystalline multicomponent oxides with controlled particle size. Similarly, hydrothermal synthesis leads to control of nanopowder morphology.

Molecular-based technologies focus on the preparation of the nanostructured mesoscale powders directly from the gas or solution phase. One molecular-based vapor-phase technology is pulsed laser deposition in which ceramic material is vaporized by a laser and vapor species are deposited on a substrate. This technology has been used primarily for thin-film preparation. Another uses combustion flames or chemical vapors to deposit molecular species directly onto a rotating or stationary substrate (Glumac et al., 1999). Sol–gel processing, another molecular-based synthesis route, is treated separately.

It is also possible to hybridize various methods. For instance, a surface pretreated via pulsed laser deposition can be used to seed a nanostructure for a sol–gel powder. Combinations of hydrothermal synthesis and microwave heating have been tried with promising results.

Nanopowders derived from vapor- and solution-phase sources can be processed in both dry and wet environments. Dry nanopowder assembly processing encompasses spray deposition, fluidized bed, and fused deposition of ceramics. Spray deposition methods include thermal spraying and cold gas dynamic spraying. Thermal spray methods are suitable for powders that can endure the high temperatures in the flame or plasma. Cold gas dynamic spraying projects particles at supersonic velocities and at low temperatures, so that particles form bonds with a surface upon impact. This is effective for nanopowder agglomerates with ductility. Another low-temperature dry nanopowder assembly process is magnetically assisted fluidized-bed deposition. In this process, magnetic forces fluidize magnetic particles, which in turn fluidize the desired nanopowders. This process is very effective in coating large numbers of objects in a very uniform fashion.

Wet nanopowder assembly processing affords the capability to disperse nanoparticles as singlets, something that cannot be achieved with dry nanopowder assembly processing. There are three requirements for effective nanopowder assembly processing: deagglomeration, dispersion, and particle assembly formation. For deagglomeration, processes such as eccentric rotating cylinder shearing, impinging jets, and ultrasonic mixing are important technologies (Riman, et al., 1998; Gelabert, et al., 2000). Dispersions based on a variety of polymer chemistries provide stability for nanopowder suspensions. To control the positions of nanopowders in suspension, another approach is polymer self-assembly [see, e.g., Brinker et al. (1999), Agarwal et al. (1997)].

Densification efforts focus on developing methods for controlling crack formation and porosity while maintaining a nanoscale grain size. Conventional solid-state and liquid-phase sintering lead to densification of nanostructured unfired mesoscale coatings at low firing temperatures. In one case, a metastable ceramic nanopowder has been densified, by exploiting a pressure-induced phase transformation, to produce a fully dense nanocrystalline material, where the final grain size of the stable phase is comparable to or less than the particle size of the starting powder. This was demonstrated for alumina and titania using low temperatures ($\sim 0.4\ T_m$) and high consolidation pressures (up to 8 GPa).

One final method, relevant also to layered manufacturing, applies advanced laser rastering methods to densify nanopowders in two dimensions. Unlike previous work with laser sintering, the laser beam is designed to incorporate a temperature distribution that induces, according to the patterned scanning of the laser, densification normal to the surface in a continuous, defect-free fashion. This leads to rapid densification times, localized heating, and the capability of changing temperature quickly allows densification of complex, fine-scale patterned structures. Thus, assemblies of nanopowders can be densified and the remaining unfired materials can be removed by various means such as ultrasonic cleaning.

Layered Manufacturing

What is layered manufacturing or solid freeform fabrication (SFF)? In recent years, the cost of making machine tools or dies to make a prototype has become exorbitant, and an alternative has been found that allows the preparation of the part without dies, thus the name "freeform." Using a computer-aided design program, a solid object is cut into layers and a computer file is created that can direct the building-up of the object layer by layer [see, e.g., Bandyopadhyay et al. (1997), Song et al. (1999)]. One approach to solid freeform fabrication is ink-jet printing. Borrowing the concept that is popular for creating written text, an ink-jet printer can print the object layer by layer, to create three-dimensional solids. In the form that this printing technology relates to ceramics, the required steps are (a) the ink preparation, (b) the ink delivery through a nozzle, (c) the break up of the ink stream into droplets, (d) the rastering of the nozzle over the substrate, (e) the registry of successive layers, and (f) the consolidation of the layers and sintering of the object to a fully dense ceramic.

Typical dimensions of each layer are $\sim 0.5\ \mu$m. The finest nozzle is about 75 μm, although micropen nozzles are being developed. A typical ink formulation contains dispersant, binder, and plasticizer with 30 vol% ceramic powder. The ink is forced through the nozzle and broken into droplets usually with electrodes. The proper placement of the drops is critical to producing the desired dimensions and aspect ratio in the object. Typically, the objects call for hundreds of layers. Following the buildup of the object, the problems with drying and firing are no different from those using more conventional processing and binder burnout.

Fused deposition of ceramics (FDC) is another useful technology for making nanocomposites. This process uses a filament of inorganic particles dispersed in a polymer as a means to deliver a molten stream of the composite onto a substrate. The droplets can be as small as 125 μm and can be deposited as multilayers for thicker coatings with a spatial positioning precision of $\pm 2.5\ \mu$m.

Current instrumentation can process as many as four different filaments to allow for the deposition of several materials on the same substrate.

To make nanostructured mesoscale coating depositions, we need to expand the applications of ink-jet printing and FDC. These technologies should be useful not only for deposition on a flat surface but also for administering multiple components. This enables the assembly of neighboring components as well as layered components of different nanostructures. Like fused deposition processing, computers equipped with fast processors can be used to assemble complex patterned structures for multifunctional systems.

Thus, one can envision multifunctional systems consisting of nanostructured capacitors, resistors, conductors, and even patterned power sources. Because the structures are computer generated, a large number of unique structures are possible.

Overall, the benefits of layered manufacturing include the ability to fabricate complex shapes, fabrication without molds, automated fabrication, and near-net shape processing. The potential drawbacks are the cost of the binder needed in the initial layering, the dimensional accuracy, the binder burnout, and the transferability of a binder delivery system to a variety of ceramic materials. When performance is more important than cost, then layered manufacturing used for a low-volume product or small number of units may be competitive with conventional processes such as forging, investment casting, or machining.

3.3 New Precursors

Polymer Precursors

What are the advantages of using preceramic polymers or precursors that pyrolyze to ceramic? The advantage is the ability to use polymer forming methods before carrying out the pyrolysis. The polymers are available as liquids, resins, and thermosets. A typical preceramic polymer would be polycarbosilane, which decomposes to SiC plus excess carbon and gaseous by-products such as hydrogen and hydrocarbons [see, e.g., Wynne and Rice (1984), Blum et al. (1989), Wu and Interrante (1992), Greil (1995)]. Other preceramic polymers contain Si–Si (silane), Si–N (silazane), or Si–O (siloxane) bonds. There are suitable preceramic polymers for aluminum, zirconium, and titanium carbides, nitrides, borides, and phosphides as well. The pyrolysis atmosphere may be nitrogen, ammonia, argon, or air. Typically, the ceramic product is porous. Nevertheless, suitable precursor chemistry and heat treatment can lead to dense materials. Another benefit of the chemical approach is the high purity achievable and the control over stoichiometry. In some cases, there are fewer steps involved with a preceramic polymer than with powder processing. For example, the pyrolysis can lead to a porous perform that requires infiltration by reaction bonding (as in RBSN, reaction bonded silicon nitride), chemical vapor deposition (CVD), or chemical vapor infiltration (CVI).

Sol–Gel Processing

Of all of the advanced ceramic processing methods, why has sol–gel processing become so prevalent? When sol–gel processing emerged about 20 years ago, there were many that thought it was a laboratory curiosity without much future.

The cost of raw materials was frequently raised as being prohibitive. Some 20 years later, the interest in sol–gel processing has not diminished. Perhaps, the reason is that there are many applications for sol–gel processing, where conventional processing does not work as well, if at all. Certainly, in optical materials, ceramic membranes, and coatings, there are many outstanding examples [see, e.g., Klein (1988, 1994)].

The sol–gel process refers broadly to room temperature solution routes for preparing oxide materials [see, e.g., Brinker and Scherer (1990), Pierre (1998)]. In most cases, the process involves the hydrolysis and polymerization of metal alkoxide precursors of silica, alumina, titania, zirconia, as well as other oxides. The solutions of precursors are reacted to form irreversible gels that dry and shrink to rigid oxide forms.

Twenty years ago, when the sol–gel process enjoyed resurgence in interest, the emphasis was on the duplication of conventionally prepared ceramics and glasses. Trying the sol–gel process was motivated by claims of the purity of the starting materials and the generally lower temperatures for processing. Clearly, the advantages of sol–gel processing outweigh the additional cost in enough cases that the skeptics are satisfied for the moment.

In today's terminology, sol–gel processing is a form of nanostructure processing. Not only does the sol–gel process begin with a nanosized unit, a molecule, it undergoes reactions on the nanometer scale resulting in a material with nanometer features. The concept behind the sol–gel process is that a combination of chemical reactions turns a homogeneous solution of reactants into an infinite molecular weight oxide polymer. This polymer is a three-dimensional skeleton surrounding interconnected pores. Ideally, the polymer is isotropic, homogeneous, uniform in nanostructure, and it replicates its mold exactly and miniaturizes all features without distortion. The nanostructure and the nanophase porosity in gels are, in fact, the features of interest, both scientifically and technologically.

The sol–gel process involves initially a homogeneous solution of one or more selected alkoxides. Alkoxides are the organometallic precursors for silica, alumina, titania, zirconia, among others. By far the most common system is one composed of tetraethyl orthosilicate [TEOS–$Si(OC_2H_5)_4$]–water–alcohol. Eventually, the solution reacts to a point where the molecular structure is no longer reversible. This point is known as the sol–gel transition. The gel is an elastic solid filling the same volume as the solution. Alkoxides react at different rates according to the electronegativity of the cation.

Most studies of the sol–gel process deal with a single alkoxide. Each precursor has its own reaction rate and complicated interdependences of pH, concentration, and solvent. Even in the relatively straightforward case of alumina, using aluminum-sec-butoxide (ASB), the expected reactions (1–4) are:

$$\mathrm{Al(OC_4H_9)_3 + H_2O = Al(OC_4H_9)_2(OH) + C_4H_9OH} \tag{1}$$

$$\mathrm{2(Al(OC_4H_9)_2(OH) = 2(AlO(OH)) +} y\mathrm{C_4H_9OH} \tag{2}$$

$$\mathrm{2(Al(OC_4H_9)_2(OH)) + 2H_2O = 2\ Al(OH)_3 + 2C_4H_9OH} \tag{3}$$

$$\mathrm{AlOOH\ or\ Al(OH)_3 = Al_2O_3 +} z\mathrm{H_2O} \tag{4}$$

A catalyst is used to start reactions and control pH. The reactions are first hydrolysis to make the solution active (reaction 1), followed by condensation polymerization (reaction 2) along with further hydrolysis. These reactions increase the molecular weight of the oxide polymer (reactions 3 and 4) resulting in either the monohydroxide AlOOH (boehmite) or the trihydroxide $Al(OH)_3$ (bayerite). Nonaqueous sol–gel processes have been used to prepare most transition-metal oxides. In systems with multiple valence, the intermediate species are oligomers that exist on the nanometer scale.

Aqueous colloidal sols also are used for sol–gel processing, recognizing that the mechanism for accomplishing the sol–gel transition is quite different. In sols such as Ludox, changing the pH or changing the concentration causes the aggregation of sol particles. The sols can be gelled in such a way that the oxide skeleton is a continuous linking of sol particles. These are discrete features that make up the skeleton corresponding to the dimensions of the sol. The other features are pores within secondary particles and pores between secondary particles. The chemical and structural differences between nonaqueous alkoxide precursors and aqueous sol precursors become blurred at later stages of the sol–gel process.

Mixing as a first step applies to the single alkoxide, multiple alkoxide, and colloidal sol processes. Absence of light scattering is a good indication of uniform mixing. Since the building blocks are nanometer in size, and smaller than the wavelength of visible light, it is easy to follow, in a qualitative way, the progress of the linking of building blocks. Gelling is often determined empirically as the time when the solution shows no flow. This is referred to as the time to gel. For this step, viscosity is a good diagnostic for the transition from a viscous liquid to a rigid structure.

Table 2 summarizes the shapes available by the sol–gel process. All of the options are more or less porous materials. In all cases, it is important that the porosity remain interconnected, whether the form of the material is essentially one-dimensional such as a fiber, two-dimensional such as a film, or three-dimensional such as bulk monoliths. These shapes can be divided into those that show isotropic shrinkage from the preform to the final form and those that show anisotropic shrinkage. Bulk materials fall into the isotropic category. Thin films on substrates fall into the anisotropic category. High aspect ratio fibers also show anisotropic shrinkage. Sol–gel thin films are exceedingly simple to prepare. A solution containing the desired oxide precursors is applied to a substrate by spinning, dipping, or draining. The process is able to apply a coating to inside and outside of complex shapes simultaneously. The time to gel is short, indicating film formation, drying and creation of pores must be rapid. When it comes to dip coatings, 50 to 500 nm coatings are easy to make but thicker coatings

Table 2 Shapes Achieved by Sol–Gel Process

Shape	Composition	Typical Application
Thin film	Titania–silica	Interference Filter
Membrane	Alumina	Ultrafilter
Fiber	Alumina–zirconia–silica	Reinforcement
Bulk		
Xerogel	Silica	Lens
Aerogel	Silica	Thermal insulation

are more difficult. Similarly, fibers can be drawn out of low-water-content solutions. The sol–gel process allows one to bait and draw a string of gel about the same diameter as the desired fiber directly from the solution.

The sol–gel process based on casting and molding can make bulk objects. The process can be used to make a microporous preform that is near-net shape. This preform is called a monolith to refer to its continuity. Monolithic gels can be formed from a colloidal sol or from an alkoxide solution. The main difference between colloidal gels and alkoxide gels is their pore structures. Alkoxide gels have small pores ($<$10 nm), while colloidal gels have bigger pores or voids between particles. Monolith fabrication is arguably the most challenging demonstration of the sol–gel process.

Having selected geometry and designed the formulations accordingly, there are several further steps common to monoliths, films, and fibers. First of all, the gels must be dried. For monoliths, drying is more difficult because of the thicker dimensions. Keeping in mind the nanostructured character of the material, several drying treatments have been developed. One possibility is aerogels that are dried in an autoclave by hypercritical techniques. That is, the solvent is removed above its critical point. The resulting gel is about 10% dense and shows no shrinkage. The more common case is xerogels that are dried by natural evaporation. Xerogels are 60% dense and have reductions 40–70% in volume.

Following reacting, gelling, and drying, gel materials have many of the characteristics of the corresponding ceramic oxide, but they are more or less porous. Interconnected pores, which remain open at the surface until the gels are fired to temperatures well above 600°C, allow the water and solvent to escape.

When the goal of the sol–gel process is a pore-free dense oxide, the final stage of processing is sintering. The high surface area of gels is looked at as a high driving force for sintering, so sintering is likely to occur at lower temperatures than in conventional powder compacts. This drive to remove porosity in the end produces a material similar to conventionally processed materials.

Remember that high purity and uniform nanostructure are trademarks of the sol–gel process. The challenge of the sol–gel process is to exploit the nanostructure aspects of the process to derive real benefits. In single-component, single-phase materials, by far the most important nanophase property is the porosity. Porosity also means that there is surface area. Sol–gel processing has a special contribution to multiphase material design and fabrication, in that the porosity provides access to the nanoscale for processes such as liquid or vapor infiltrations, physical or chemical depositions, and chemical reactions such as pyrolysis or oxidation/reduction.

Because the sol–gel process is a low-temperature process, it is possible to incorporate organic material. This is accomplished by infiltrating a previously formed oxide gel with monomer, creating an organic–inorganic copolymer with a metal alkoxide, or simultaneously polymerizing monomer and metal alkoxide. Because of the scale of mixing of the organic and inorganic phases, these materials are nanocomposites. The products of some of these syntheses can be classified as sequential interpenetrating networks or simultaneous interpenetrating networks.

Among the features of sol–gel processing, the one that should stand out at this point is its nanometer scale. In the long run, the advantages for the sol–gel approach to nanophase ceramics materials are the simple processing steps, the flexibility of solution chemistry, the low-temperature treatments, and small investment in equipment.

When considering where sol–gel materials are used, most of the applications are substitutions of a sol–gel material for a ceramic obtainable by other means. Nevertheless, there are some applications that are unique to sol–gel processing, such as porous materials for membranes or infiltration and organic–inorganic hybrids.

4 SUMMARY AND PROJECTIONS

In the end, the choice of what method to use depends on many factors. According to the SP^3 paradigm, performance is the ultimate test of synthesis and processing. Therefore, choices are made on the basis of how easily the process can be optimized for chemical properties or physical properties. Other considerations are the availability of the appropriate raw materials or precursors and the availability of the processing equipment. The advanced processing schemes in many cases require high-temperature furnaces or reactive atmospheres. Difficulty gaining access to this equipment or conditions may prohibit the use of an advanced process. Similarly, problems of scale-up in size or number of components may make a conventional process more straightforward than an advanced process. Finally, time and cost are factors in the decision whether or not to use an advanced process. Nevertheless, there continue to be signs of progress in all of the advanced processes described, with some, such as microwave processing, in more widespread use than others, such as layered manufacturing. As with any new technology, change is slow, but competition from other materials is a strong incentive for advanced processing of ceramics.

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PART 7

APPLICATIONS AND USES

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CHAPTER 36

SPACECRAFT APPLICATIONS OF ADVANCED COMPOSITE MATERIALS

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1 INTRODUCTION

Many rigorous demands are placed on the design of spacecraft structures. They must be lightweight to maximize the allowable payload mass while being stiff enough to maintain precision alignment of instruments under severe orbital environmental conditions. Launch loads often dictate strength requirements that are much greater than those needed for space operation. In today's economic environment the time and funding available for project completion are reduced, and more requirements are being placed on the designer. Advanced composite materials are being used more frequently and in more diverse applications to assist in meeting these demands, such as those shown in Fig. 1. Effective use of advanced composites often requires that the analyst, designer, and fabricator work together to reduce costs, optimize structural performance, and minimize mass.

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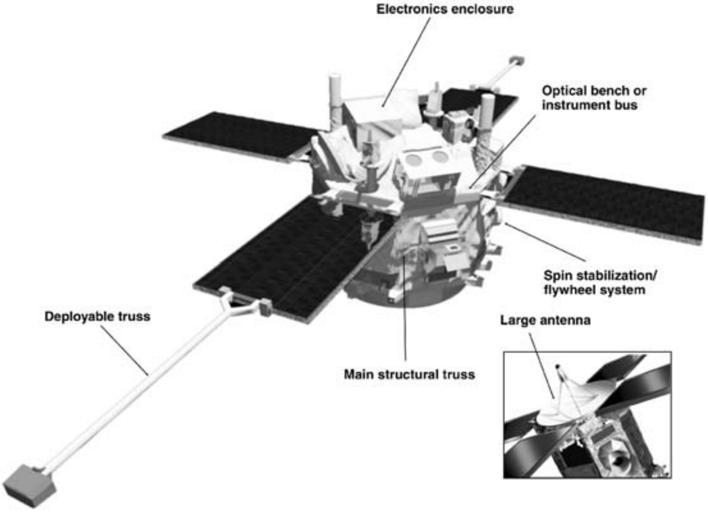

Fig. 1 Common applications of advanced composites on spacecraft structures.

This chapter consists of three sections. Section 1 briefly describes the basic characteristics and properties of advanced composites. Section 2 focuses on the specific properties of advanced composites that are important for spacecraft operation. Examples of typical spacecraft structures constructed in part or entirely from advanced composite materials are given in Section 3.

1.1 Advanced Composites

A composite material is generally defined as a man-made material composed of two or more distinct components. The term *advanced composites* generally refers to materials consisting of high-stiffness continuous-fiber reinforcements within a comparatively weak matrix. The reinforcing fibers are generally much stronger and stiffer than the matrix and are the primary drivers for the unidirectional tensile and compressive stiffness and the strength of the material. The primary role of the matrix is to provide a load transfer mechanism between fibers. When fibers and matrices are combined to form a composite material, the result is a material with properties that are more than simply the sum of the individual components. Properly selected fiber and matrix combinations complement each other and work together to form materials that have unique and valuable properties.

Advanced composites consist of several thin plies (lamina) stacked together to form a laminate. The individual laminae (plies) are orthotropic and transversely isotropic, with the strength and stiffness values in the fiber direction being an order of magnitude or greater than the corresponding values in the direction transverse to the fibers. Each ply within the laminate can be oriented at a different angle to create a laminate that meets the structural requirements desired while using the minimum amount of material. Additionally, plies of different materials can be combined into one laminate, or individual plies can be hybridized to obtain the best material combination.

The constitutive stress–strain equations for composite laminates are much different than those for isotropic materials. For laminates the traction (tensile/compressive) forces per unit width are coupled with the moments per unit width. Classical laminated plate theory is used to describe the behavior of composite laminates assuming plane stress conditions (Eqs. 1 and 2):

$$|N_i| = |A_{ij}||\varepsilon_j^0|| + |B_{ij}||\kappa_j| \tag{1}$$

$$|M_i| = |B_{ij}||\varepsilon_{ij}^0| + |D_{ij}||\kappa_j| \tag{2}$$

In this set of equations, $i, j = 1, 2, 3$ and N_i are the in-plane tensile/compressive forces per unit width on the laminate, M_i are moments per unit on the laminate, A_{ij}, B_{ij}, D_{ij}, are the constituent matrices, and ε_{ij}^0, κ_j are the reference-plane strain and laminate curvature. The moments and the forces are coupled through the bending-coupling stiffness matrix. In its current form, as tensile forces are applied, the laminate will warp due to the coupling. If the B matrix is identically equal to zero, the extension and bending equations become decoupled, therefore physical bending and extension are independent of each other. The laminate can then be designed (through proper use of fiber orientations in the lamina) to take advantage of this coupling/uncoupling to produce the desired effects. The A, B, and D matrices relate the lamina properties to the laminate properties by way of the material properties and laminate thickness. An example is given below for the calculation of extensional stiffness matrix, A_{ij}:

$$A_{ij} = \sum_{k=1}^{n} (\overline{Q_{ij}})_k [h_k - h_{k-1}] \tag{3}$$

where n = the number of plies (lamina), and h_k = the distance from the top of the kth ply to the laminate reference-plane; $(\overline{Q_{ij}})$ is the ply-transformed stiffness matrix, which directly relates to the material properties of the ply in laminate axes. An example is shown below for the calculation of the transformed matrix:

$$\overline{Q_{11}} = Q_{11}m^4 + 2(Q_{12} + Q_{66})m^2n^2 + Q_{22}n^4 \tag{4}$$

where m and n are the direction cosines in the transformation and the ply stiffness (Q_{ij}) values are represented by:

$$Q_{11} = E_{11}/(1 - \nu_{12}\nu_{21}) \tag{5}$$

$$Q_{22} = E_{22}/(1 - \nu_{12}\nu_{21}) \tag{6}$$

$$Q_{12} = E_{11}\nu_{12}/(1 - \nu_{12}\nu_{21}) \tag{7}$$

$$Q_{66} = G_{12} \tag{8}$$

where E, G, and ν are the familiar elastic modulus, shear modulus, and Poisson ratio for the different material directions (longitudinal or transverse to the fibers) in each of the plies. Several texts are available that describe classical laminated plate theory and composite mechanics in detail, such as *Mechanics of Composite Materials*.[1]

Reinforcement Fibers

The majority of reinforcement fibers belong to one of four families: carbon fibers, aramid (aromatic polyimide) fibers, glass fibers, and specialty exotic fibers. Special reinforcement fibers such as boron and alumina fibers are sometimes used for niche applications but are not widely used due to a variety of reasons such as low availability, high cost, and nonstandard handling requirements. In spacecraft applications, carbon fibers are used far more frequently than aramid or glass because they have the best combination of specific strength, specific stiffness, cost, and handling characteristics. There are, however, some applications where aramid or glass fibers are better suited, such as those where thermal isolation, corrosion resistance, or toughness are critical design factors.

Glass fibers were the first fibers to be used extensively as reinforcements in composite materials. They are relatively inexpensive, widely produced, and frequently used today in applications where low cost is a primary requirement, such as in the automotive and recreation industries. Different grades of glass fibers are available, the most common being E-glass, which provides good performance at moderate prices. S-glass fibers provide higher strength and stiffness than E-glass but at a significantly higher cost. Although glass fibers have been used extensively in aerospace applications, they have been replaced by stiffer and higher strength carbon and aramid fibers in modern high-performance aerospace applications.

Aramid fibers, such as DuPont's Kevlar fiber, were introduced commercially in the early 1970s.[2] Filament wound pressure vessels and lightly loaded secondary aircraft structures were early applications for aramid fibers. A unique feature of these fibers is that they are remarkably damage tolerant. The tensile failure of aramid fibers is characterized by fibrillation and a large amount of energy can be absorbed before material failure. This property makes aramid fibers especially useful in situations where properties of energy absorption or dispersion are critical, such as micrometeoroid impact protection.

Carbon fibers are the most frequently used reinforcement in high-performance composite materials today. A broad selection of carbon fibers encompassing a range of strength, stiffness, thermal, and electrical properties is available. Carbon fibers are grouped into two families according to the precursor material from which they are made: (1) synthetic polymer fiber precursors and (2) pitch-based fiber precursors. The first carbon fibers were produced by pyrolysis of rayon in 1959 by Union Carbide. In the early 1960s, mass-produced fibers appeared, produced from the pyrolysis of PAN (polyacrylonitrile), a synthetic precursor. Less expensive carbon fibers made from a pitch base have been produced since 1973.[3] PAN fibers offer higher strength, stiffness, and improved manufacturing consistency over pitch-based fibers. Consequently, they are the fibers of choice for most high-performance composite applications. On the other hand, refinements in the production of pitch-based fibers have produced carbon fibers with much better thermal and electrical conductivity than is possible in PAN fibers. Although these advanced pitch-based fibers still do not equal the strength and stiffness of PAN fibers, they are indispensable for applications that require good thermal conductivity, such as electrical enclosures.

To ease manufacturing and improve the bond between the fiber and the polymer matrix, all reinforcement fibers are available with various surface treatments. These treatments include epoxy sizing for carbon fiber, silane coupling agents for glass fiber, and plasma etching for aramid fiber surfaces. Reinforcement manufacturers work closely with their customers to select or develop the appropriate surface treatment for optimization of material behavior for a given application.

Polymer Matrices

The majority of current advanced fiber-reinforced composite materials employ thermoset or thermoplastic polymers as the matrix material. Table 1 lists some properties of common polymer matrix materials used in advanced composites.

Thermosetting polymers are characterized by chemical cross-links between molecular chains. These cross-links are formed during the resin curing process and are primarily responsible for the mechanical properties of the cured polymer. Thermoplastic polymers do not have chemical cross-links, and their rigidity depends on the mechanical interaction of highly intertwined molecular chains. Properties of new high-performance thermoplastics match or exceed many traditional thermosetting polymer properties, but these are often more difficult to use because of the very high temperatures and pressures required.

Thermosetting epoxies have been the dominant matrix material for advanced aerospace applications due to the wide-ranging combination of mechanical properties, processing and handling characteristics, and variety of formulations available. Lately, polycyanate thermoset matrix materials are replacing epoxies because they are much more durable in the space environment (see discussion in Section 2.1 on environmental durability). Advanced thermoplastics have been used in some space applications, but their use has been limited due to processing complexity.

Many aspects must be factored into the polymer matrix selection for a particular composite material application. The material properties that are important for composite material applications differ from application to application and can include service temperature range, environmental durability, toughness, strength, stiffness, compatibility with fiber sizing, availability, processing characteristics, and cost.

1.2 Unique Benefits of Advanced Polymer Matrix Composites

Advanced fiber-reinforced composite materials have several key properties that make them especially useful in spacecraft applications. Their high specific

Table 1 Properties of Common Polymer Matrix Materials

Matrix Material	Tensile Strength (MPa)	Tensile Modulus (MPa)	Service Temperature (°C)
Epoxy 1	50	3100	140
Epoxy 2	90	3800	245
Polyester 1	75	3380	90
Cyanate ester 1	80	3600	240
Thermoplastic	100	3900	150

strength and stiffness (strength/density, stiffness/density, examples are given in Table 2) allow spacecraft structure designs that are lighter than traditional metal structures. Structural mass may be reduced by more that 50% over comparable metal structures through the use of advanced composites. Because the total mass of a spacecraft is limited, the minimization of the structural mass of the spacecraft can extend the spacecraft life by allowing larger fuel loads, or more mass may be allotted for scientific payloads.

While simply replacing metal components with quasi-isotropic composite laminates can result in reduced mass, the exploitation of the directional nature of advanced continuous fiber composite materials results in designs that are much more structurally efficient. A structure made from isotropic metals is generally sized according to Von Mises loading principals. This often leads to structures being dramatically overdesigned in the nonprinciple loading directions. Composites are much stronger and stiffer in the direction parallel to the fibers (longitudinal) than the direction transverse to them. Designers can take advantage of this directional strength to produce structures with equal margins of safety for all loading directions. In this way, the use of anisotropic composite materials leads to a more efficient, lower mass design.

Another extremely useful trait of advanced composites is that the thermal expansion properties can be customized. Carbon and aramid fibers have a *negative* axial coefficient of thermal expansion (CTE), so composite structures using carbon fiber or aramid fiber reinforcements can be designed that have ultralow thermal expansion. Spacecraft are typically subjected to a wide temperature range as they move in and out of direct solar radiation. The use of metallic structures in this environment can lead to substantial dimensional changes that disturb instrument alignment and induce stresses. The use of composites with customization of the coefficient of thermal expansion permits the design of precision structures and assemblies that function uniformly over the whole temperature range.

1.3 Scope

The aim of this chapter is to discuss the benefits of using advanced composite materials in spacecraft structures and to provide examples of typical applications. Advanced composites can consist of a wide variety of matrix and reinforcement materials. Because it is impractical to discuss the full range of possible materials and their applications in this text, the discussion will be limited to the most widely used advanced composites, specifically polymer matrices with long fiber reinforcements. The materials that will be discussed are carbon, glass, aramid fiber reinforcements, and typical polymer matrix materials such as epoxies and

Table 2 Comparison of Specific Properties of Metals and Composites

Material	Specific Tensile Strength	Specific Tensile Stiffness
AL 2024-T3	174	26
AL 6061-T6	115	26
Ti-6Al-2Nb-1Ta-1Mo	184	25
T-600 Stainless Steel	127	25
Quasi-isotropic Gr/Ep	258	50

cyanate esters. The following paragraphs will briefly mention some of the more common applications that will not be discussed in detail in this chapter.

Advanced composites have a long history of use in spacecraft launch vehicles (generally rockets). Various parts of rocket propulsion systems have been made with composite materials. For example, rocket motor cases that make use of the directional properties of advanced fiber-reinforced composites are stronger and lighter than metallic motor cases. Also, rocket engine nozzles using ablative polymer matrices are significantly lighter than metal nozzles and offer improved temperature stability. Other launch vehicle components, such as aerodynamic shells and fairings, are frequently made from advanced composite materials for mass reduction.

Heat shields for atmospheric entry constitute a unique application of composite materials. Long fiber reinforcements in composite heat shields give them the ability to maintain form and shape during ablation produced by the heat of atmospheric entry.

2 USE OF ADVANCED FIBER-REINFORCED COMPOSITES IN SPACECRAFT

Advanced fiber-reinforced composite materials have a long history of spacecraft use. The primary reason for their use is to reduce the mass of spacecraft structures and components. Additionally, their unique high specific strength and stiffness can be utilized to create structures that are impossible with conventional metal construction. This section addresses the basic characteristics and properties of advanced composite materials that are important for spacecraft applications.

2.1 Properties/Characteristics for Space Applications

All material properties that can affect component performance must be considered during design, but the relative importance of each varies for different types of components. For example, the design of structural truss members must include criteria such as total mass, thermal stability, environmental durability, and stiffness. On the other hand, electromagnetic shielding and thermal conduction are critical for the design of electronics enclosures.

Coefficient of Thermal Expansion

Because of the anisotropic nature of advanced composites the coefficient of thermal expansion (CTE) of composites can be altered to meet a range of design specifications. In contrast to most structural materials, carbon and aramid fibers have a *negative* axial CTE. Therefore the CTE of composite structures is not a fixed value as for metals but can be selected by the designer. Realistic carbon composite structures can and have been produced that have a near-zero or even negative CTE. Because spacecraft temperatures on orbit can range between extremes of +120 to −90°C as the spacecraft passes in and out of Earth's shadow, this near-zero CTE provides stability unmatched by metal structures.

Typical CTE values for high modulus carbon fibers and aramid fibers are -0.9 and -2.7 $\mu\varepsilon/°\text{C}$, respectively. Unidirectional polymer matrix composites made using these fibers then have longitudinal CTEs ranging from -0.7 to -0.4 $\mu\varepsilon/°\text{C}$ for carbon fiber composites, and -4.0 to -2.7 $\mu\varepsilon/°\text{C}$ for aramid fiber composites. For individual lamina, the CTE value transverse to the fibers is governed by the matrix properties and typically ranges from 20 to 35 $\mu\varepsilon/^\circ\mathrm{C}$. Most applications of composites have plies oriented at various angles, producing a net CTE in between the longitudinal and transverse values, depending on the specific layup. Shown in Fig. 2 are values for the CTE of carbon fiber/epoxy composite laminates for various layups. For laminates with plies at various angles, fiber stiffness significantly affects the total laminate CTE since the reinforcement fibers are much stiffer than the polymer matrix. In general for angle ply laminates, as fiber stiffness increases, the net laminate CTE will decrease. This is a major reason why ultrahigh modulus fibers are often selected for precision spacecraft components.

Although the low CTE of composite laminates is an extremely valuable characteristic, it can lead to new design problems, such as CTE mismatches in metal-to-composite bonded interfaces. Careful attention must be given to thermal strains to prevent bond failure or part deformation. Furthermore, because spacecraft often use a combination of composites and metals, the thermal expansion of the complete structure must be analyzed to ensure that stress levels and deformations stay within allowable limits over the entire temperature range.

High Specific Stiffness and Strength

The most common reason for using advanced composites is mass reduction. Unidirectional high modulus carbon fiber composites can have a longitudinal stiffness greater than 550 GPa. This is 8.5 times greater than the specific stiffness (stiffness/density) of most engineering metals, all of which have approximately the same specific stiffness (see Table 2). Because unidirectional laminates have low transverse strength and stiffness, they are not commonly used in real applications. Instead, laminates containing plies oriented at various angles are designed according to the principle and secondary loads. Although the specific

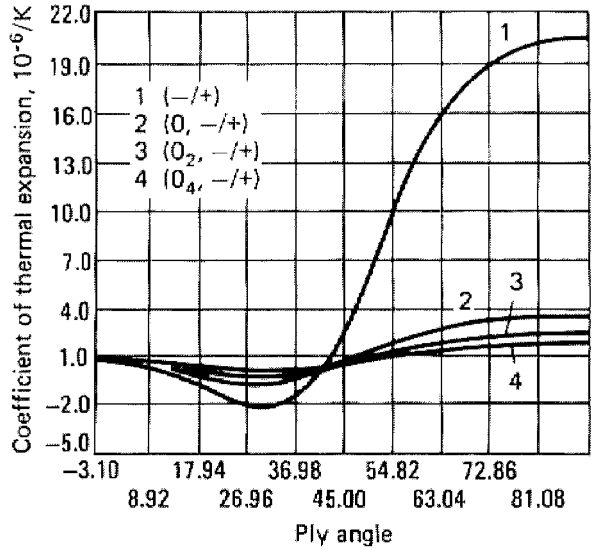

Fig. 2 Longitudinal CTE for typical graphite reinforced laminates.

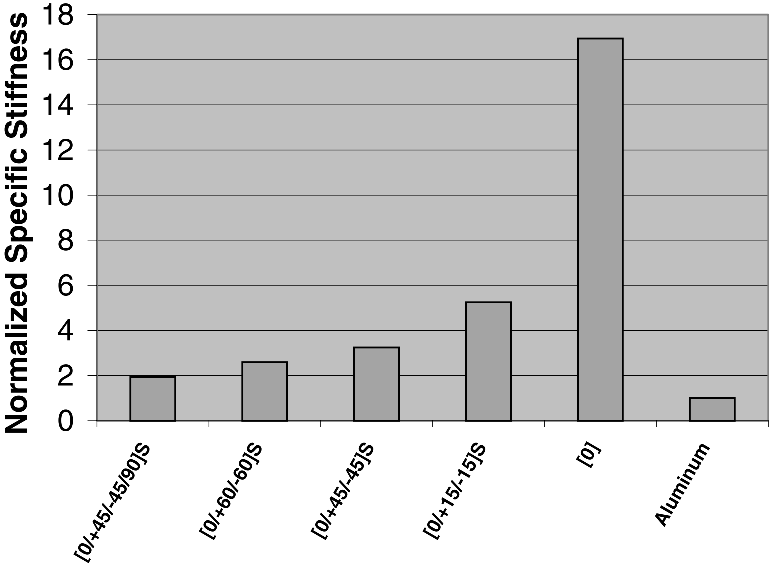

Fig. 3 Comparison of specific stiffness of various IM7/8552 laminates normalized to specific stiffness of aluminum.

strength and stiffness of angle-plied laminates is less than that of unidirectional laminates, it is still superior to metals. Shown in Fig. 3 is the specific stiffness of IM7/8552 carbon fiber/epoxy laminates for various lay-ups. Note that since the specific stiffness is greatest for a unidirectional lay-up, careful selection of lay-up is key to achieving maximum mass savings.

Thermal Conductivity

Recent interest in using composites for electronics enclosures has led to higher thermal loads being placed on composite structures. In fact, in electronics enclosure applications for spacecraft, the thermal demands are often more important than mechanical considerations.[4] Modern pitch-based fibers that have thermal conductivity exceeding that of aluminum have been developed to help meet these new demands (Table 3).

The thermal conductivity of composite laminates is similar to their mechanical characteristics in that they are highly directional and sensitive to matrix selection

Table 3 Comparison of Typical Pitch Fiber Thermal Conductivities with Aluminum[5]

Fiber	Fiber Conductivity (W/m°C)	Quasi-isotropic Laminate Conductivity (W/m°C)
Amoco K1100	1100	322
Mitsubishi K13C	640	188
NGF YS-90A	500	151
Amoco P55	120	37
Aluminum	167	167

and lay-up. For typical carbon fiber composites, the transverse and through-the-thickness thermal conductivity is three to four orders of magnitude less than the longitudinal thermal conductivity. Although this is often viewed as a disadvantage of composites, it can be exploited to design specific thermal paths within component modules.[5] Often a quasi-isotropic lay-up is chosen to simplify thermal and mechanical analysis, but they are generally *not* optimal for structural efficiency. Unfortunately, analytical prediction of the thermal conductivity properties for various laminates has proven difficult and quasi-isotropic lay-ups are therefore selected to simplify design.

EMI Shielding and Electrical Characteristics

Electromagnetic interference (EMI) shielding is an important concern for most space flight programs. Shielding mitigates the effect of high-energy solar radiation and is used to isolate instruments from one another. Conventional EMI shields are made of metals such as aluminum and tantalum. Many studies have been done evaluating the use of composite materials for electronics enclosures with the primary aim of reducing the total mass of the enclosures while maintaining acceptable EMI shielding levels.

A typical shielding level required for spacecraft is 60 dB for a frequency range from 0.10 to 18 GHz.[6] Although carbon fibers are inherently electrically conductive, laminates with carbon fiber reinforcements generally do not provide adequate EMI shielding when used alone. Several approaches to increase the level of shielding have been studied by various groups. Common approaches to increase the shielding effectiveness include the addition of metal foil or mesh to the laminate, coating the finished laminate with metal, or the use of metal-coated fibers within the composite plies.[7]

Many research groups have measured the shielding effectiveness of carbon fiber-reinforced composite laminates. The standard test method follows MIL-STD-285 or some variation thereof. Although this standard was originally intended to test the shielding effectiveness of enclosures used in EMI testing, it is widely used as a procedure to test materials, gaskets, ventilation panels, and apertures. The test procedure consists of placing a radiation source and a receiving antenna on opposite sides of a shielded room with a shielded barrier between them. The material to be tested is placed over an aperture in the barrier and the signal strength at the receiving antenna is measured. The signal loss is then taken as the shielding effectiveness.

Test results consistently indicate that the shielding characteristics of unaugmented carbon fiber composites are much more similar to metals than to plastics. By adding solid metal laminations or coatings to composite laminates, the shielding effectiveness is roughly equivalent to that of solid aluminum. For multipiece shield assemblies, experimental testing has shown that joint surface contact resistance is critical for shielding effectiveness. Consequently, nickel and silver are sometimes selected over aluminum or copper because of their superior oxidation resistance.

Recently, highly conductive polymers have been investigated to increase the shielding effectiveness of composites and eliminate the need for metal coatings.

Additionally, research into using high conductivity boron interculated graphite fibers (boron placed into the interstices of the graphite fibers) has produced promising results.[8]

Environmental Durability

The impact of space environmental effects on materials is dependent on the type of mission, and more importantly, the orbit in which the spacecraft operates.[9] The orbital space is generally divided into three regions based on orbit altitude: low-Earth orbit (LEO, up to 1000 km), mid-Earth orbit (MEO, 1000–35,000 km), and geosynchronous Earth orbit (GEO, 35,000 and higher). The types and intensities of various environmental effects depend greatly on orbit altitude and inclination. In this section the effects of various environmental factors on advanced polymer matrix composite materials will be discussed, each of which produce distinct effects on materials. The relative importance of each of these effects is a function of the orbital placement and must be evaluated for each mission.

The predominant environmental factors influencing spacecraft are (1) atomic oxygen, (2) ultraviolet radiation/solar exposure, (3) micrometeroid and debris impact, (4) thermal cycling induced microcracking, (5) contamination, (6) vacuum-induced outgassing, (7) spacecraft charging, and (8) environmental synergistic effects. Of these, atomic oxygen, vacuum-induced outgassing, and thermal cycling–induced microcracking are of unique concern to polymer matrix composites.

The following discussion of space environmental effects on composites is based on Silverman,[9] and the reader is referred to it for further explanation.

Atomic Oxygen Effects. Atomic oxygen erodes organic materials causing surface recession and degradation of optical and thermal properties. Experimental results from the long-duration exposure facility (LDEF) clearly demonstrate that atomic oxygen in LEO will erode all polymeric materials, including those commonly used on spacecraft for thermal and electrical insulation, as paint vehicles, and as composite matrix materials. The rate of erosion varies for different materials, and in some cases is a function of exposure time. Although there is no simple way to predict the susceptibility of certain materials to atomic oxygen erosion, a broad database has been collected that can be used for preliminary material screening. Of particular interest is data that confirms that polycyanate matrices have much lower atomic oxygen reactivity than epoxy matrices.

The amount of surface recession due to erosion is directly proportional to the atomic oxygen fluence (total integrated flux), hence the recession on a particular surface is dependent on its location on the spacecraft and the surface attitude relative to the flight path, spacecraft altitude, orbit inclination, and solar activity conditions. In selecting materials for spacecraft, the designer must be aware of atomic oxygen effects and calculate the total amount of surface erosion that will occur. If this value is considered unacceptable, protective coatings that have low atomic oxygen reactivity may be indicated. Materials successfully used as coating materials include extremely thin metals, silicon oxide, aluminum oxide, and silicone room temperature vulcanization (RTV).

Vacuum-Induced Outgassing. Polymer matrix composites tend to outgas due to moisture desorption or evolution of volatiles within the material. The expelled gases can be especially detrimental to optical instruments and solar cells aboard the spacecraft. Surfaces may be obscured by condensed outgassing products or local clouds may be formed that affect sensitive instrument readings. Additionally, molecular contamination from outgassing can degrade the performance of thermal control surfaces, especially those that rely on passive radiative cooling.

Outgassing of composites is characterized by three quantities based on ASTM E595: total mass loss (TML), collected volatile condensable material (CVCM), and water vapor regained (WVR). CVCM is a measure of the potential for outgassed products to form surface deposits, while TML is a measure of the potential for the formation of molecular clouds. Typical current design requirements are $<1.0\%$ TML and $<0.1\%$ CVCM, but these are expected to become more stringent for future spacecraft. Outgassing data on specific material systems is available in several databases. Of particular importance is the data indicating that polycyanate and thermoplastic matrix materials characteristically produce less outgassing products than conventional epoxy matrix materials.

Thermal Cycle–Induced Microcracking. Thermal cycling–induced microcracking is due to the difference in the CTE of each individual ply parallel to and transverse to the fiber direction. The CTE parallel to the fibers is dominated by fiber properties and can be slightly negative. The transverse CTE is controlled by the matrix properties and is generally about half of the value for the CTE of the matrix alone. Consequently, internal stresses are induced due to differential thermal expansion for any composite lay-ups that have plies oriented at different angles. During repeated thermal cycles cracks form and grow parallel to the fiber direction of each ply.

Microcracking of composites can cause strain hysterisis and significant changes in the overall CTE, both of which present dimensional stability problems. However, microcracking is sometimes employed to achieve a desired CTE. For example, the struts on the metering truss of the Hubble space telescope were deliberately subjected to microcracking to “tune” them to the desired CTE.[10] New toughened epoxies and polycyanate matrix systems have greatly reduced the severity of microcracking, but designers should remain aware of the potential for thermal cycling–induced microcracking.

2.2 Typical Structures

Several “standard” structural forms are used for composite spacecraft structures: solid laminates, sandwich laminates, strut structures, and isogrid structures. Each form has benefits and restrictions ranging from cost to performance. This section will discuss these forms and their applications.

Solid Laminate Construction

Solid composite laminates are the simplest of the forms used in composite structures. Several plies of a selected composite material are stacked to create a final laminate of the desired thickness. The laminate may be treated as a continuous, homogenous material once its properties are determined. As such, structural analysis is simplified and uncertainties are reduced. Solid laminates are best suited to applications where the primary loading is in-plane tension or shear. Examples include primary structural members that must withstand the axial launch and maneuvering loads, and torsion tubes such as those used to mount deployable solar cell arrays and antennas.

Sandwich Structures

In applications where bending stiffness and strength are critical, sandwich structures are often used because they provide high bending strength and stiffness with low mass. Sandwich structures consist of two (or more) facesheets separated by a low-density core (Fig. 4). This configuration provides substantially greater bending strength and stiffness with little mass increase when compared to the facesheets without cores. Sandwich panels with aluminum facesheets have been used in a wide variety of aerospace applications, providing much higher bending stiffness and strength values. The desirable properties of advanced composites such as low coefficients of thermal expansion and directionality also apply to sandwich structures with composite facesheets.

The most commonly used core material for space applications is aluminum honeycomb, although other core materials such as aramid honeycomb and syntactic foam are sometimes used. Typical honeycomb cores for space applications range from 12.7 to 50.8 mm in thickness, and cell size ranges from 3.18 to 12.7 mm. Important properties that affect the selection of various types of sandwich structures are core venting, core CTE, and the need for discrete hardpoints that are used to attach the panel to the structure and to mount instruments to the panel.

Venting. A very important consideration that must not be overlooked is that the core must allow venting of gases within the core. If this is not done, high pressure inside the core can lead to facesheet debonding or core explosion in the space vacuum environment. The cell walls in aluminum honeycomb are usually perforated for venting requirements.

CTE. Thermal expansion characteristics of the core must also be considered for successful sandwich structure utilization. The in-plane coefficient of thermal expansion (CTE) value is usually provided by the manufacturer and is assumed

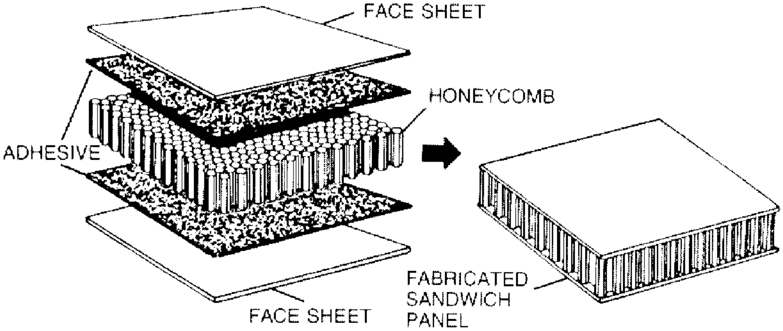

Fig. 4 Sandwich structure construction. (Figure reprinted with permission of the Society of Manufacturing Engineers, *Fundamentals of Composites Manufacturing: Materials, Methods, and Applications,* Copyright, 1989.)

to be constant for all in-plane directions. However, recent experience has shown that the CTE of aluminum honeycomb core may be different in the ribbon and fill directions, therefore caution must be used when designing dimensionally stable sandwich structures. Additionally, the through-the-thickness CTE is dictated by the material that the honeycomb is composed of (e.g., aluminum) so careful attention must be given to the design of end close-outs and fittings to ensure compatibility with the sandwich structure.

Hardpoints. Because the facesheets in sandwich structures are very thin, discrete hardpoints must be integrated into the sandwich panel to provide locations for mechanical fasteners. The hardpoints prevent fastener pullout and localized panel crushing by distributing the mechanical loads over a larger area of the panel. These hardpoints are most often individual metal inserts bonded to the sandwich panel. While the need for hardpoints introduces additional complexity and expense, careful planning for fastener locations and fastener quantities will minimize the increased fabrication cost.

Truss Structures

Truss structures composed of many individual struts are common in spacecraft to provide stiff, large structures with low mass. Advanced composites are ideally suited for strut applications because they carry predominantly uniaxial loads. This allows the directional properties of fiber-reinforced composites to be exploited, producing a very efficient structure. The advantages of composite use for mass reduction are augmented by the thermal stability inherent in composite truss structures, which can be ensured by the specification of a wide range of values for the coefficient of thermal expansion. A typical composite strut is shown in Fig. 5. Aluminum or titanium end fittings are generally bonded to the composite strut to provide attachment points.

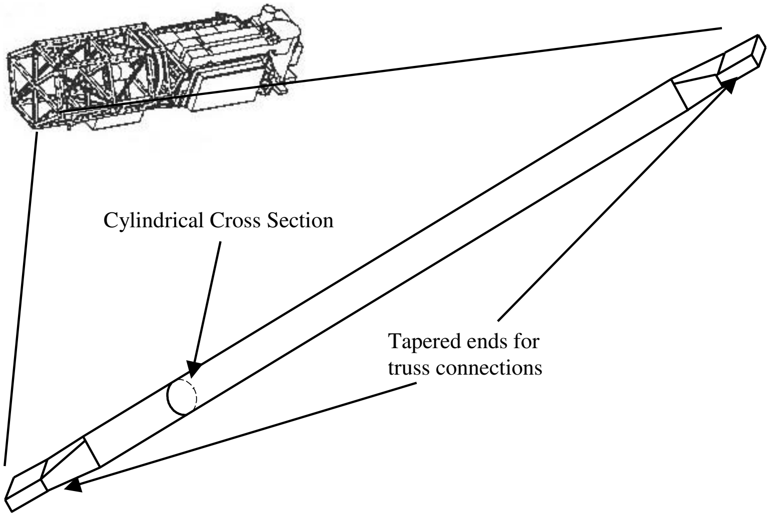

Fig. 5 Typical composite truss member.

Isogrid Structures

Isogrid construction is a new alternative method of fabricating structures that have high bending strength and stiffness at low mass. Originally developed for compression-loaded metallic panel designs, the isogrid concept incorporates a unique triangular pattern of stiffener elements instead of a core structure to achieve required stiffness.[11] Several new programs have been initiated to fabricate advanced composite materials structures using the isogrid concept.

An isogrid stiffened panel is composed of three sections, each constructed from the same base composite material. The three sections are the primary facesheet, the web, and the flange stiffeners, as shown in Fig. 6. Because isogrid stiffened panels have only one complete facesheet, more radiating area is exposed than in conventional sandwich panels. Consequently, isogrid stiffened structures offer improved heat dissipation over that of honeycomb core structures. The additional radiating area is especially advantageous in the design of solar arrays where the efficiency of the array is strongly dependent on operating temperatures. Isogrid construction has the potential to reduce the cost of complex structures through reduced tooling and assembly costs.

Joints

The design of composite structures must include joining considerations. Laminated composites tend to have poor bearing strength and toughness when compared to metals. Consequently, bonded joints are strongly preferred over the use of discrete mechanical fasteners for joining composite materials. In contrast to joints using discrete mechanical fasteners, bonded joints provide a distributed load transfer that greatly reduces stress concentrations. The performance of bonded or bolted composite material joints for a variety of loadings and geometries has been evaluated in many studies.[12] Results indicate that adhesively bonded joints offer superior strength and reliability when compared to joints that use mechanical fasteners such as bolts and rivets.

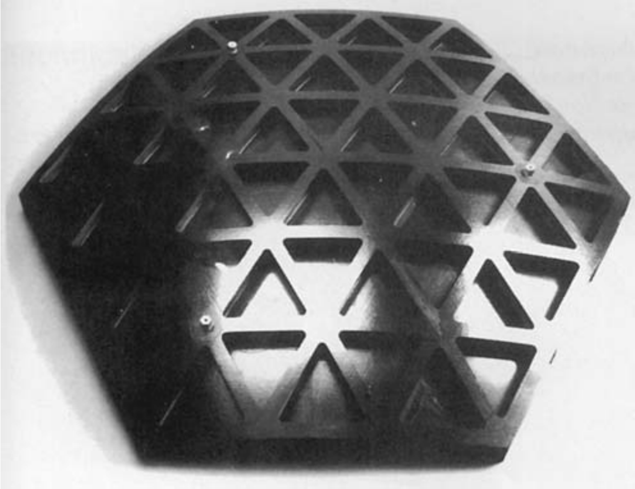

Fig. 6 Isogrid stiffened panel (reprinted from Ref. 11).

2.3 Manufacturing

No discussion of the application of composite materials is complete without an overview of the wide range of manufacturing methods available. In fact, the manufacturing process is integral to the effective and cost-efficient use of composites and must be considered throughout the entire design process. The manufacturing method that is best suited to the application may be dictated by a combination of total cost constraints, production time, production quantity, and the physical properties required by the designer. It is impossible to provide a concise description of all possible manufacturing methods here. A comprehensive text such as *Fundamentals of Composites Manufacturing* provides specifics on the various composite manufacturing methods/processes.[13] Some of the unique aspects of composite structure manufacturing will be discussed here, with a primary focus on cost reduction.

Reduction in component cost and complexity are always welcome attributes for spacecraft. Because composites are built up and formed by material addition instead of material removal, consolidation of many individual components into one entity can be achieved. This consolidation reduces the part count (and therefore cost) and results in more compact and efficient structures. Reduction of the size and mass of entire systems has become more important as spacecraft become smaller. Part consolidation is a key technology to achieving this goal.

New communication and observation systems that use constellations of multiple small satellites are being designed. Spacecraft design for these programs represents a departure from the traditional design philosophy because many similar or identical satellites must be produced in a costly manner. The use of advanced composite structures instead of metals may potentially reduce recurring manufacturing costs significantly. Most metal parts for spacecraft are produced by the removal of material from a larger entity to achieve the finished form. This machining constitutes the majority of the cost in the production of metal parts. The use of composite components on the other hand can drastically reduce recurring machining cost and material waste because a single reusable mold can be used to produce many near-net shape parts.

An assortment of cost reducing automated or computer-assisted processes are available that may minimize labor expenses. Commonly used manufacturing processes include filament winding, automated tow placement, automated ply cutting, laser-projected lay-up templates (guides), pultrusion, matched die molding, and resin transfer molding. Not all processes work equally well for every structure; instead one or two processes are usually far superior for a particular structural configuration.

Multifunctional Structures

Mechanical parts, sensors, wiring, and electronic components can be combined in single composite structures to form integrated, or multifunctional, structures. Because composite materials are manufactured or built up from many thin individual plies, individual component elements may be embedded within the layers as they are applied (referred to as packaging). The resulting multicomponent integration is a necessary design element in the success of future small satellite missions. The embedding of passive or active electronic components provides new opportunities and venues for integrated component diagnostics, sensing, and structural health monitoring.

Multifunctional structures are broadly defined as structures that support additional tasks that may be unrelated to basic mechanical load carrying.[14] Examples of functions included in this definition are damping enhancement elements, passive electronics and circuits, sensors, actuators, and active electronics. Multifunctional structures may include different levels of complexity that range from simple passive thermal management or electromagnetic waveguides to advanced fully integrated adaptive structures containing sensors, actuators, and active electronics. Research groups have commonly adapted a stepping stone approach toward the achievement of the ultimate goal of highly integrated structures with minimum mass and volume. The vast body of work in this area will not be covered here, but a brief overview of some of the more mature technologies is provided below.

Embedded-Sensor Technology. Substantial work has been done to develop techniques for embedding sensors in composite structures and in testing their response. Much of this research has been focused on the embedding of various types of strain sensors in composite structures to create integral, real-time distributed sensing of local strains. The data provided by these sensors is useful for the determination of the status of deployable structures, in the design of the shape or form of spacecraft components, in the measurement of vibration levels, and for damage detection.

Starting in 1994, a program of research began into remotely queried embedded microsensors (RQEM) sponsored by the Naval Research Laboratory. RQEM developed a working prototype of the sensor and solved many of the difficult research issues. In 1997 a follow-up program, Applied Research in Remotely-Queried Embedded Microsensors (ARRQEM), refined the system concept (Fig. 7) and solved many of the difficult engineering issues.

The primary target materials for the installation of remotely queried sensors were the various aerospace composites, including glass and carbon-fiber reinforced thermoset and thermoplastic resins. It is undesirable to use "wired" sen-

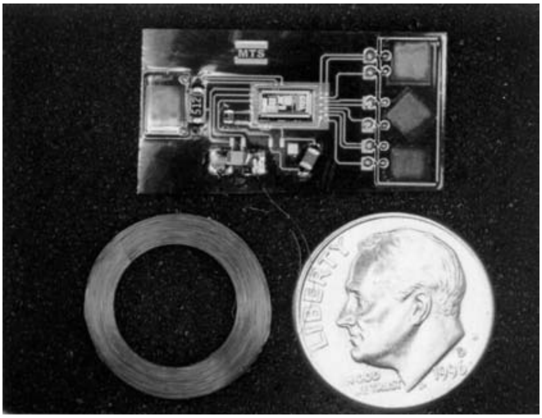

Fig. 7 ARRQEM sensor package.

sors (strain gauges, optical-fiber-based grating sensors, etc.) on these materials due to difficulties in placing the sensor and maintaining the connecting wire or fiber during composite fabrication. For wired sensors, the connection requires slits or holes in the layers, or it must extend across the layer to the edge of the lay-up. Either method is tedious to produce and introduces flaws or potential flaws into the composite material, because structural integrity is compromised. Flaws can be minimized through the use of very fine wires, but they are delicate and often break during lay-up, which is also a problem in the use of optical fiber. To avoid these difficulties, the U.S. Office of Naval Research (ONR) funded development programs for wireless sensors that can be powered and queried without physical connections.

ONR had several high-level requirements for the ARRQEM sensor system. The primary requirement was the measurement of various physical phenomena (e.g., strain, pressure, temperature) within a composite structure. Other requirements included:

- Low recurring cost (including installation costs and the use of consumable components such as sensors)
- Usable lifetime commensurate with the instrumented structure (50 years in some cases)
- Interrogation possible from a minimum distance of several inches without physical contact
- Accommodation of a varying number of different or similar types of sensors in close proximity
- No significant effect on the instrumented structure (e.g., do not exceed the critical flaw size)
- Correlation of the measured effects with true effects in the observed structure
- Safety in handling and use during structure processing and service life

Several of these requirements (e.g., lifetime, flaw size, safety) preclude the use of embedded batteries.

In the program an embeddable strain gauge rosette (shown in Fig. 8) was successfully developed and limited testing was conducted. Further development in this area will lead to the application of this technology to satellites and other complex composite structures.

Integrated Electronics. Traditional electronic systems designs provide for one or more printed wiring boards (PWBs) that may be surface mounted on primary structures or enclosed by a separate structural housing. Although this method is simple and time tested, mass and volume are not minimized because PWBs are usually nonstructural elements. Composite materials can be used to create printed wiring boards that function as both electrical *and* structural components. This then eliminates the need for a separate support structure, i.e., the housing, leading to drastic reductions in the mass and volume of electrical systems—an important factor for small spacecraft design.

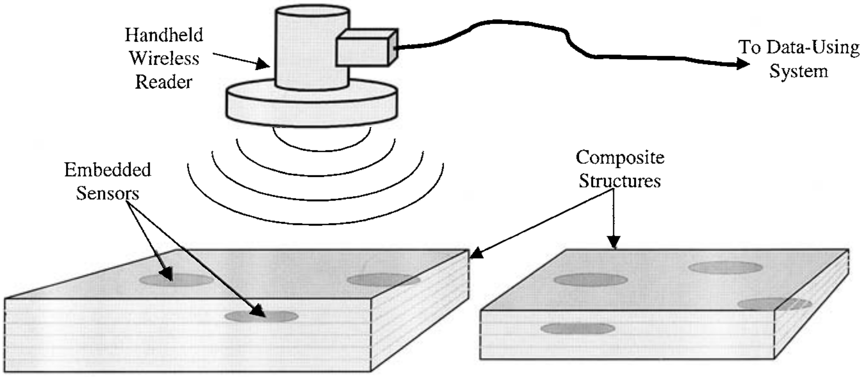

Fig. 8 ARRQEM system concept. Wireless interrogation of inexpensive embedded sensors are conducted by external reader device. Digitally-represented sensor readings are transferred to the device for storage or analysis.

3 EXAMPLE SPACECRAFT APPLICATIONS

Because the total allowable mass of complete spacecraft systems is limited by the launch vehicle constraints, the minimization of structural mass is required to maximize the allowable payload mass. This minimization can lead to the extension of spacecraft operating life because more fuel can be carried, or additional scientific payloads can be accommodated. Use of advanced composite materials is a key factor in reducing the structural mass of spacecraft but also provides other advantages such as low thermal expansion and manufacturing flexibility. Table 4 shows the most common spacecraft structures and the relative importance of structural properties to each.

Specific applications and examples of composite material use in space structures will be described in the following sections. The space structures are classified as either primary or secondary structures, and are further grouped by specific component classes. For spacecraft, primary structures are those that are critical to achieve the basic mission, for example, surviving launch and on-orbit

Table 4 Considerations in Spacecraft Material Selection

Property	In Truss Members	In Primary BUS	In Deployable Structures	In Electronics Enclosures	In Optical Benches	In Mirrors/ Reflectors
Mass	√√√	√√√	√√	√√√	√√	√√√
CTE thermal stability	√√	√√	√√	√	√√√	√√√
Thermal conductivity	√	√	×	√√√	√	√
Electrical conductivity	√	√	×	√√√	√	×
Cost	√√	√√	√	√√	√	×
Stiffness	√√√	√√	√√√	√√	√√√	√√√
Strength	√	√	√√√	√	√	√

Note: ×: not critical, √: somewhat important, √√: very important, √√√: mission critical.

station keeping maneuvers. Secondary spacecraft structures are any that are related to the performance of specific mission functions, such as instrument support structures. The information in this section is intended to provide an overview of the existing state of the art of composite spacecraft structures and to address the associated advantages and limitations.

3.1 Primary Spacecraft Bus/Chassis Structure

Advanced composites are being used more frequently for primary spacecraft bus structures to reduce mass and increase precision in large, one-of-a-kind spacecraft and smaller high-production spacecraft. Typical designs employ multiple struts that support stiffened panels that serve as decks for mounting instruments. Examples of both types are described below. Issues associated with the use of advanced composites for primary spacecraft bus structures will also be addressed.

Example: FORTE

An example of modern, low-cost, low-mass composite spacecraft primary structures is the fast on-orbit recording of transient events (FORTE) spacecraft, developed jointly by the Los Alamos National Laboratory and Composite Optics Inc.[15] The compressed development schedule of this small spacecraft dictated a simple, modular design with a minimum number of unique parts. Bulk fabrication methods may be efficiently employed for the production of multiple parts, and cost and complexity are reduced. Because the same tooling is used to produce multiple units, the resulting parts are dimensionally consistent and thus interchangeable.

The spacecraft structure consists of three decks and three octagonal frame cages (trusses). The decks are composed of carbon fiber/epoxy laminates and aluminum honeycomb core that are bonded to form a sandwich panel. Copper foil is co-cured onto the external surfaces of the sandwich panels to provide positive grounding and EMI shielding. All of the sandwich panel facesheets are 0.762 mm thick and have a ply layup of $[0/60/120]_S$. Aluminum-threaded inserts are bonded into each deck arranged with hole patterns and hardware sized specifically for each component. A total of 25 different components are mounted on the three decks.

For simplicity and expedience, all of the frame parts are cut out of 1.22-mm-thick $[0/45/90/135]_S$ flat carbon fiber/epoxy laminates. A computer-controlled waterjet cutter was used to cut out the frame parts from two large flat laminates. The parts were then “snapped” together using mortise and tenon joints. Finally, the parts were placed in tooling fixtures and bonded together. The use of this streamlined manufacturing process provides rapid and repeatable fabrication of the structures and eliminates the need for multiple unique and expensive tools.

The original design of the FORTE spacecraft specified an all-aluminum structure. The use of composite materials for this design led to a mass reduction of approximately 50% over the aluminum structure. Additionally, the low-mass composite design improved the inertial properties of the FORTE spacecraft. Finally, because of the utilization of advanced design and manufacturing technology, the cost of the composite structure was comparable to that of the equivalent aluminum structure.

Example: Midcourse Space Experiment

The Midcourse Space Experiment (MSX), a large sensing platform, has precise pointing and tracking requirements that are critical for mission success.[16] The spacecraft is divided into three sections: an electronics platform/launch vehicle interface, a thermally stable truss section, and a temperature-controlled instrument section. The truss section forms the center of the spacecraft and provides the primary support for the Space Infrared Imaging Telescope III (SPIRIT III). Stiffness, rotational stability, minimization of mass, low coefficient of thermal expansion, and low thermal conductivity were critical design requirements that could only be satisfied by the use of composite materials for the truss members.

The truss section is shown in Fig. 9. It consists of 20 carbon fiber/epoxy I-beams connected with titanium fittings and carbon fiber/epoxy plates. Three different sizes of I-beams are used, classified according to their location: lower diagonals, upper diagonals, and verticals. Customized titanium fittings join the I-beams, provide mounting interfaces for the SPIRIT III instruments, and connect the truss section to the upper and lower spacecraft sections.

Carbon fiber/epoxy gusset plates are used to join the upper and lower diagonals (Fig. 10). Gusset plates are commonly used for planar joints of composite or metal members. When composite beams and/or gusset plates are used, adhesive bonding is the preferred method of joining rather than the use of me-

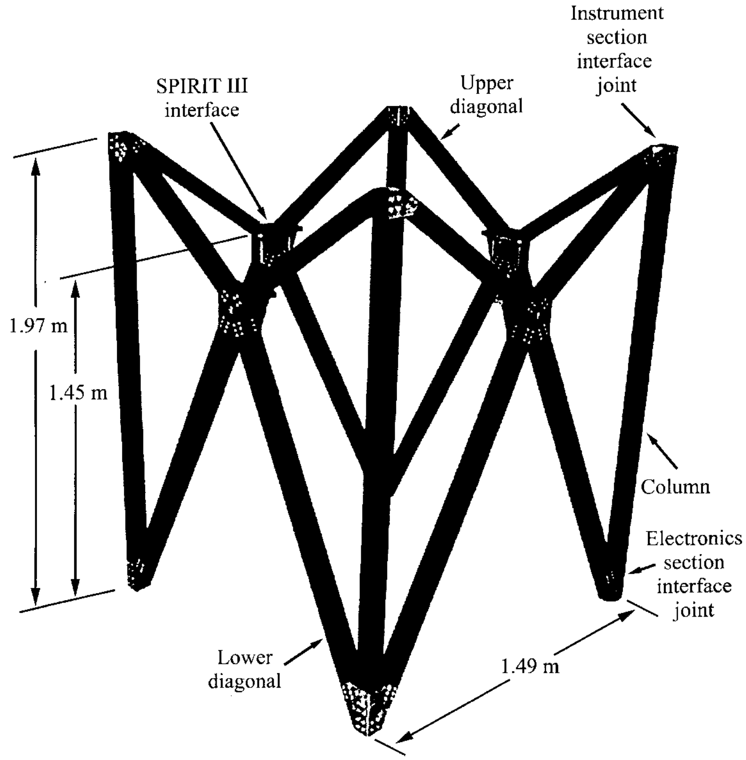

Fig. 9 Composite strut structure of MSN spacecraft (reprinted for Ref. 16).

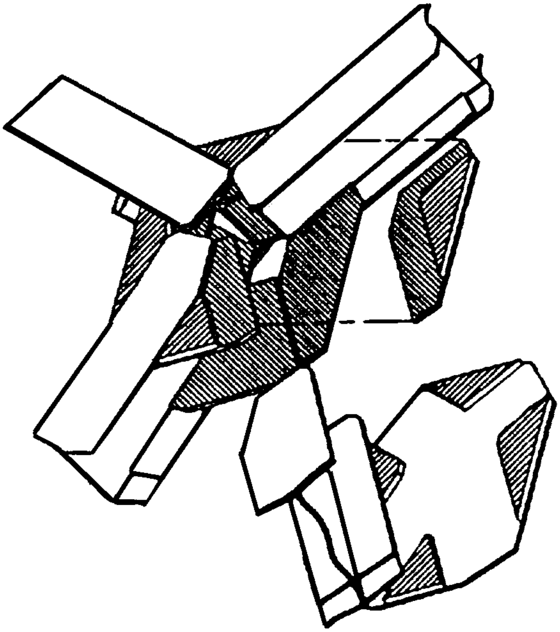

Fig. 10 Example of typical gusset plate joint.

chanical fasteners. This is an important difference between laminated composite structures and metal structures, and one that must be considered for successful design.

The carbon fiber/epoxy system selected for the MSX I-beam construction was T50/ERL1962. T50 carbon fiber reinforcement was selected for its high stiffness values coupled with minimal thermal conductivity. The ERL1962 resin (Amoco) was selected because it is a toughened epoxy system that exhibits good resistance to microcracking. The I-beam ply lay-up, $[0_2/60/-60]_{S3}$, results in maximum axial stiffness. Prior to assembly an eutectic coating was applied to control moisture uptake, and the beams were then painted with Chemglaze Z306 flat black polyurethane paint. The total mass of the truss assembly is 47.1 kg.

3.2 Deployable Structures

Deployable structures are defined as those that can be stored in a small volume configuration for launch and that are later deployed for space operation. Deployable structures are required elements for many missions to accommodate the need for large spans or planar components that are impossible to launch in their extended configuration. The most common deployable structures in use today are solar arrays, which are folded for launch. The recent delivery of the additional solar panels to the International Space Station is an example of this small-volume launch to large-volume expansion. Current and future missions will require deployable structures to support large baseline optical instruments, space-based radar applications, experiments that require environmental isolation, or solar sails.

Deployable structures can be classified as either *rigid* or *inflatable*. Inflatable structures require hydrostatic (gas) pressure for deployment, in contrast to rigid deployable structures that use purely mechanical means for deployment. Inflatable structures may be subdivided into three groups: continuously inflated structures, rigidized inflatable structures, and single inflation structures.

Inflatable Structures

When compared to rigid deployable structures, inflatable structures generally have smaller packaged volume and lower mass. This technology is a significant contributing element in the design of future large solar power systems.[17] There are three categories of inflatables, which are abbreviated as follows: continuously inflated (CI), rigidized inflatable (RI), and single inflation (SI).[18] CI structures require gas pressurization over the duration of their service life. Inflatable rafts used for recreational water sports are a typical example of CI structures. RI structures require inflation for initial deployment support only until the structure is rigidized, usually via planned material property changes. SI structures are designed for one-time, usually short-duration use, e.g., the airbag system for the Mars Pathfinder Lander.

Both the mass of the structure itself and the mass of the deployment gas, together referred to as the ‘‘wet’’ mass, must be considered in the spacecraft design process. Since rigidized inflatable structures generally have the lowest values for wet mass they are the best choice for most inflatable applications. Consequently, the majority of inflatables research has been dedicated to RI development. Some applications, however, such as habitation modules, short-duration experiments, and landing systems are best solved by utilizing CI or SI structures.

Continuously inflated structures must remain pressurized throughout their entire service life. Some benefits include form definition and simplicity of technology required. Since a pressurized membrane is consistently taut, CI structures have better surface definition than rigidized inflatables. This surface definition is particularly useful in antenna and reflector applications. Additionally, a CI structure is technologically simpler than a RI structure because no material transformation is required for operation. However, sufficient inflation gas must be supplied for the entire operational life span of the mission, and this requirement affects the mass parameters of other mission elements. The total quantity of gas required varies according to several factors, e.g., inflation pressure, estimated gas leakage, total inflated volume, and mission life span. CI structures are therefore not ideal for long-term or long life-cycle applications because mission operation would require a prohibitively large amount of inflation gas.

Rigidized inflatable structures are better suited to long life-cycle applications because only the amount of inflation gas needed for initial deployment is required. Several approaches to rigidization have been investigated, and polymer matrix composite materials appear to be the material of choice. Rigidization is usually achieved through a chemical or phase change of the polymer matrix (such as curing), which increases its stiffness and therefore the stiffness of the whole composite structure.

3.3 Electronics Enclosures

Lightweight advanced composites electronics enclosures have been developed for many different vehicle applications, including spacecraft. Electronics enclosures are discrete structures designed for the sole purpose of holding printed wiring boards and electrical components in place. Composite enclosures can be made significantly lighter than machined aluminum enclosures and may be produced at a lower unit cost while possessing equal or better mechanical and thermal performance. Three groups of functional requirements—mechanical loading and physical attachment specifications, thermal control for installed electronic components, and environmental shielding—must be satisfied in the design process for electronics enclosures.

Both physical support and protection for electronic components (often printed wiring boards) are provided by electronics enclosures. Design considerations must include the accurate alignment of printed wiring boards (PWBs) to a motherboard or input/output connection area, and provisions for mechanical attachment to the spacecraft bus. The enclosure must be stiff enough to meet internal deflection specifications during launch conditions, and alignment and support must be maintained over thermal extremes. Designs usually include a removable cover to facilitate installation, removal, and exchange of enclosed components.

Mechanical loads from launch or thermal expansion are the primary drivers for the structural design requirements. Static equivalent of dynamic loads are calculated from a curve of power spectral density of random vibration, and launch loads are generally used to represent loadings for mechanical analysis, as described by Steinberg.[19] The walls of the enclosure can then be sized by analyzing them as individual orthotropic flat plates with simply supported boundaries. This method greatly simplifies initial sizing and enables the designer to create a more optimum structure. This method is superior to the standard ‘‘black aluminum’’ design process where metals are simply replaced with dimensionally equivalent quasi-isotropic composites. Roberts[20] has shown that a reasonable, if somewhat conservative, approximation of actual static deflections and natural modes can be produced with orthotropic analysis.

The thermal environment inside the electronics enclosure must be controlled to ensure the safety of sensitive internal electronics, so some provision must be made to expel heat generated by electronic components. The thermal dissipation path may consist of several parts. Paths begin at individual electronic components, but all must end at the final heat sink or thermal reservoir, usually space. A typical thermal path may be subdivided as follows: electronic component to PWB, PWB to card guide, PWB and card guide to the enclosure wall, wall to mounting feet, and feet to base or enclosure wall to space. Conduction is the chief mode of thermal flow in electronics enclosures with the exception of radiation from the enclosure walls to space.

The composite fiber properties rather than the resin properties are the main contributors to values for in-plane thermal conductivity. Carbon fiber composites can be made that have equal or better in-plane thermal conduction than solid aluminum.[21] However, through-the-thickness thermal conduction is limited by the polymer matrix and is generally several orders of magnitude less than in-plane conduction. As such, careful attention must be given to the design of thermal attachments to ensure adequate thermal conduction across joints and connections. Metallic inserts or coatings are commonly used for thermal load distribution through-the-thickness of composite panels.[22]

Electronics enclosures must provide radiation and electromagnetic shielding to preserve and to protect the electronics within the housing. This requirement has received the majority of attention because composites have much higher electrical resistance than metals. New techniques have been developed and implemented to increase or augment the shielding effectiveness of composite enclosures. A variety of specific methods are based on the use of metallic foils, coatings, or meshes incorporated with the laminate as discussed above.

Examples

The following descriptions of composite electronics enclosure designs represent current practice and the state of the art.

A National Aeronautics and Space Administration (NASA) study (Fenske et al.[23]) described a comparison study of composite versus aluminum electronics enclosures. A multidisciplinary team produced a set of specific electrical, thermal, and mechanical requirements (summarized in Table 5) that were used as criteria for the development of three different new enclosure types. These lightweight electronics enclosures were independently developed and tested against a baseline machined aluminum enclosure. Each development team used a different manufacturing method to produce the separate enclosures. The three enclosures—cast aluminum, lightweight machined aluminum, and composite—were compared to the baseline standard machined aluminum enclosure specifically in terms of cost and performance. Thermal, electrical and mechanical specifications were satisfied by all three of the enclosures developed.

Composite Optics Inc. used proprietary SNAPSAT technology to manufacture the composite electronics enclosure. This approach produced an easily scalable design that can be used to produce enclosures housing between 6 and 14 PWBs. The enclosure (Fig. 11), consists of a double-wall system that minimizes cost and mass by limiting use of expensive highly conductive materials. Aluminum foil was co-cured onto the inner side of the walls to provide shielding but was later found to be insufficient for grounding requirements. Additional thin copper strips were later soldered directly to the nickel-plated PWB attachment fittings to achieve adequate grounding.

Table 5 lists the mass of each enclosure and the corresponding launch costs assuming a figure of $10,000/lb to orbit.

An independent research and development team at the Johns Hopkins University Applied Physics Laboratory (JHU-APL) used previous research to develop a composite electronics enclosure that could be used on a variety of spacecraft, without requiring substantial modification or redesign.[24] The project goal was to produce an electronics enclosure with the mass reduction and high stiffness benefits of composites and without high recurring manufacturing costs.

The JHU-APL electronics enclosure design (shown in Fig. 12) consists of a three-sided box with integral mounting flanges bonded to a baseplate thermal plane. Unidirectional pitch fiber contained in the baseplate thermal plane was

Table 5 Comparison of Composite and Lightweight Aluminum Electronic Enclosures[23]

Enclosure Type	Weight [kg (lb)]	% Weight Savings from Original Design	Launch Cost at $10K/lb
Original machined aluminum	8.4 (18.5)	N/A	$185,000
Lightweight machined aluminum	14.4 (9.7)	48%	$97,000
Cast aluminum	4.5 (9.9)	46%	$99,000
Composite	2.6 (5.8)	69%	$58,000

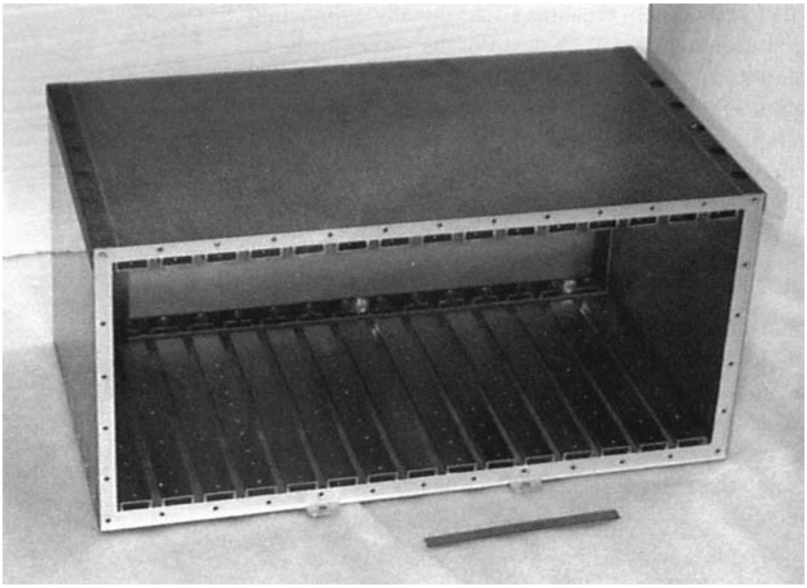

Fig. 11 Composite electronics enclosure (from Ref. 23).

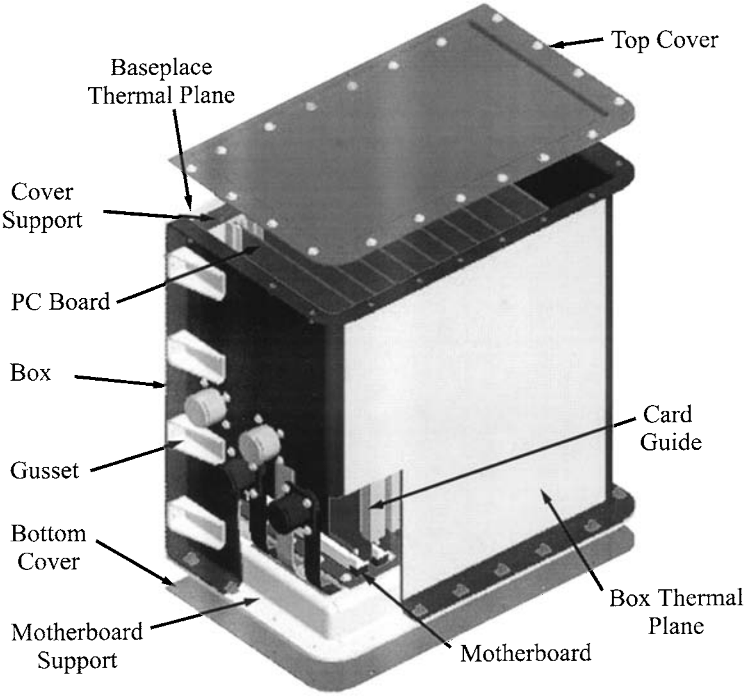

Fig. 12 Composite electronics enclosure (from Ref. 24).

used to distribute heat over the entire area, to increase thermal transfer efficiency. The outer wall acts as a secondary thermal plane and also contains pitch fiber. The total quantity of expensive high thermal conductivity pitch fiber is minimized by making the thermal path from the PWB to the spacecraft or thermal reservoir as short as possible.

Data gained from this program supports the conclusion that a composite electronics enclosure can be built with equivalent or better performance than a machined aluminum design and with lower unit cost. The simplicity of the design and tooling developed by JHU-APL make the techniques applicable to a wide range of missions. Approximately 50% mass savings can be achieved by using composite electronics enclosures over traditional aluminum designs. In a follow-up study investigating the optimization of design with respect to structural and thermal loading, it was shown that judicious discrimination in the use of expensive high thermal conductivity materials is key to minimizing the total cost of the enclosure. The two designs developed in the study had equivalent mass, thermal, and structural performance, but the difference in cost between the two was 10-fold, with only one design being equal in costs to the original machined aluminum standard.[25]

3.4 Optical Benches and Instrument Structures

The advantages in the use of composite materials can be utilized in individual instrument structures as well as the larger spacecraft structures. Optical benches that support critical instruments such as telescopes and interferometric optics, are commonly constructed from carbon fiber composites because they offer thermal stability impossible to attain with conventional metal structures. Optical bench construction usually includes one or more panels or struts that provide mounting, support, and alignment of optical instruments. Composite optical benches have been successfully used in applications that range in size from smaller than this handbook to as large as the Hubble space telescope. An overview of the fundamental requirements and basic structures that have been used for optical benches is presented here.

Typical Optical Bench Requirements

While there are differences and unique requirements for every optical bench, the elements of dimensional stability, stiffness, and mass are the three main requirements common to most designs. As previously mentioned, dimensional stability is the primary requirement for optical benches. Tight tolerances must be maintained throughout manufacturing, launch, deployment, and service life. Taken together, meeting specified tolerances is often a challenging task.

Near-zero thermal expansion (in one or two directions) is a key property of composites and one that is invaluable for optical instrument applications. Stringent dimensional tolerances are often required for the location and alignment of optical components. These tolerances must be maintained over a wide range of operating temperatures. For example, Composite Optics Inc. imposes a standard maximum angular tolerance of 30 μrad for all optical components, and even tighter tolerances are specified for more advanced instruments.[26] Stiffened carbon fiber composite panels are the material of choice to satisfy these tolerance requirements. The panels are generally sandwich structures with either honeycomb core or discrete rib stiffeners. Facesheet laminates for these panels have been designed to meet optimal stiffness, coefficient of thermal expansion (CTE), and thermal conduction requirements with minimal mass. High modulus or superhigh modulus carbon fibers are often used in facesheet laminates to achieve the desired properties.

Stiffness requirements for optical benches are usually described by specifying a minimum natural frequency, typically 30 Hz or greater. Boundary conditions (fixity) and mounting conditions must be considered during the design process.

The total mass of the bench and fittings is usually limited to less than one third of the mass of the instruments that it supports.[26]

Design and Manufacturing Considerations

Several important physical constraints are common to composite optical bench designs. Because thin composite facesheets cannot support threaded fasteners, separate hardpoints or fittings must be used. A common technique for providing component mounting points involves the postbonding of aluminum or titanium inserts into the bench sandwich structure, shown generically in Fig. 13. The insert must be bonded to both the core material and the facesheet to ensure good structural performance. In honeycomb core structures, the bonding usually requires the use of a strong paste adhesive for the insert-to-facesheet bond and a low-density potting adhesive for the insert-to-core bond. In addition to discrete inserts, metal edge close outs are sometimes used as hardpoints for attachment to the primary spacecraft structure or other instruments. The bonding of metal parts into composite structures must be done cautiously because differences in thermal expansion between composite and metals may result in large stresses within the adhesive layer, sandwich structure, and metal components.

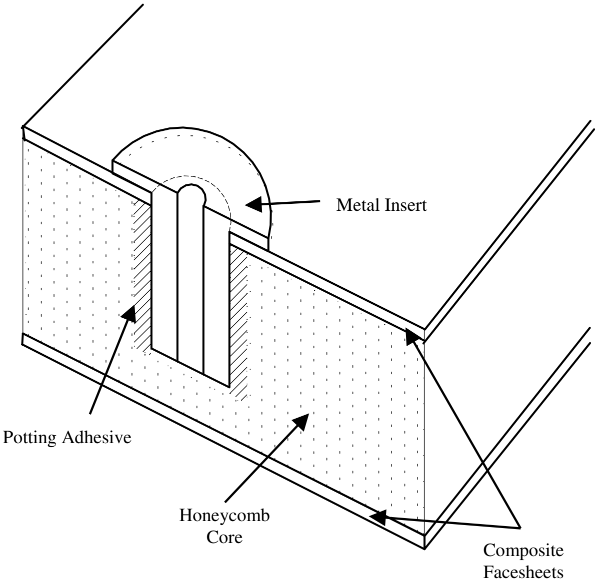

Fig. 13 Cutaway view of typical hardpoint insert in composite sandwich panel.

Outgassing and volatile evolution are factors that must also be considered when using polymer matrix composites for optical bench designs to be used in space. As discussed in Section 2.1, polymer matrix materials are subject to outgassing caused by moisture desorption or the evolution of volatiles within the material. The evolved gasses can form clouds or films that can impact the performance of sensitive optical instruments. Polycyanate matrix materials are preferred over epoxies for optical bench elements because they exhibit much lower moisture uptake and therefore less outgassing. Special preflight handling procedures such as nitrogen gas purge treatments are often specified to minimize outgassing.

When using low CTE composites for optical benches, allowances must be made to accommodate dimensional changes from thermal expansion in the mounting of metal components onto composite optical benches. For example, aluminum instruments mounted on a low CTE composite bench will exhibit dimensional changes that are much greater than those of the deck itself. Instrument mounting must be designed so these thermally induced dimensional changes do not exceed acceptable distortion or stress levels.

3.5 Antennas, Reflectors, and Mirrors

Composites have been widely used to construct low-mass antennas and reflectors. For example, the high-gain communications antenna on the *Voyager* spacecraft employed honeycomb core sandwich construction to reduce mass while retaining structural requirements. Glass or ceramics materials traditionally used for mirror construction and mirrors using these materials have areal densities of 25–30 kg/m^2.[27] Equivalent composite designs can have areal density as low as 1–3 kg/m^2 resulting in an order of magnitude mass reduction over glass and ceramic designs.

Carbon fiber-reinforced composites have additional properties that make them useful in reflector applications. These include:

- Near-zero CTE
- High structural efficiency
- High strength
- High fracture toughness
- Design and manufacturing flexibility

Proper functioning of mirrors and reflectors requires the sustained maintenance of proper form or figure during service life. Because the thermal extremes encountered by space structures are quite large, thermal stability of these structures is a critical requirement. Furthermore, mirror form and figure must also be maintained over many thermal cycles. The thermal stability criteria are easily met if the unique thermal expansion properties of advanced composites are employed.

Mirrors, reflectors, and antennas using composites fall into two groups: all-composite designs, and hybrid designs. Hybrid designs use composite support structures but combine these with noncomposite elements such as glass or ceramic reflective facesheets. All-composites designs use composite elements for all parts of the mirror, including the reflective surface. Coatings such as aluminum, silver, or gold are applied to all composite mirrors to provide a highly reflective surface. The following section describes hybrid composite mirrors and all-composite mirrors that represent the current technology.

Hybrid Composite Mirrors

Hybrid designs consisting of composite structures that support glass, ceramic, or metal clad facesheets have been used for visible wavelength applications. Although these designs weigh much more than all-composite designs, these hybrid mirrors have exhibited the best quality ambient figure. Additionally, because traditional optical finishing techniques are used to achieve the appropriate figure in these structures, fewer problems may be encountered than in all composite designs. An example of a hybrid visible mirror is the Next Generation Space Telescope Mirror System Demonstrator (NGST NMSD), developed at Composite Optics Inc.[28]

The NGST NMSD mirror (shown conceptually in Fig. 14) employs a glass front facesheet with a composite supporting structure and rear facesheet. The aperature is 1.6 m and the areal density for the mirror assembly is 15 kg/m^2, 11 kg/m^2 for the mirror substrate and 4 kg/m^2 for the reaction structure. During the design process manufacturing techniques were considered along with the physical and mechanical properties of glass, composites, and adhesives. This strategy was taken to ensure that maximum performance (low mass) was achieved at minimum cost.

Ultrahigh modulus carbon fiber/cyanate ester resin composites were chosen to construct the back facesheet and shear web core because of their high stiffness, low density, near-zero thermal expansion properties, and manufacturing

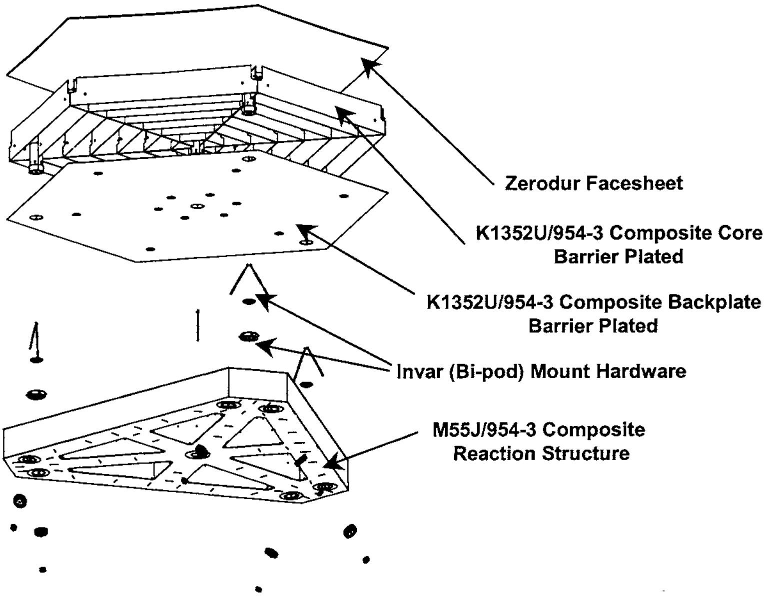

Fig. 14 NGST hybrid composite mirror (reprinted from Ref. 28).

flexibility. For the front reflective facesheet, Zerodur glass was chosen because it has a small negative CTE and can be shaped using traditional optical finishing techniques. Waterjet pocket milling techniques were used to remove material from the backside of the facesheet leaving an isogrid stiffening pattern. A toughened paste epoxy adhesive (EA 9309) was used to bond the composite shear web core to the front Zerodur glass facesheet and composite rear facesheet. Thick adherend shear tests conducted in the operating temperature range (room temperature to 35 K) proved that the adhesive would withstand the cool-down thermal strain differentials between the mirror and the core

A subscale test mirror was fabricated with the same materials, construction, and processing used in the full-scale mirror. A key concern was ensuring that the thermal expansion of the composite supporting structure was the same as that of the Zerodur reflector. Because small variations in composite characteristics are unavoidable a thin metallic plating was applied to the composite structure to fine tune its CTE. This proved to be an effective and predictable method for CTE management as no measurable change in ambient figure was detected in the test thermal ranges after 50 cycles. The development, fabrication, and testing of this mirror proved that thermal strain management may be achieved by proper material selection.

All-Composite Mirrors

All-composite reflector designs for X-ray, microwave, and radio-frequency (RF) applications have been successfully produced. An example of an all-composite design is the Microwave Limb Sounder (MLS) primary reflector.[29] An off-axis parabolic surface provides the front reflective surface shape, with a 1.6-m major axis and 0.8-m minor axis elliptical projected aperture. The structure of the MLS reflector is similar to that of the NMSD discussed previously, except the reflective facesheet is composite instead of glass. Two 24-ply carbon fiber/polycyanate composite facesheets are adhesively bonded to a triangular composite isogrid core to create the reflector. The performance requirements and the actual operational values of important reflector parameters are listed in Table 6. RF reflectivity, solar concentration, and thermal absorption requirements dictated a specialized treatment for the composite surface. An initial precision bead blasting, followed by vapor deposition coating with aluminum, and a final layer of silicon suboxide satisfied the performance parameters.

Table 6 MLS Composite Primary Reflector Performance

Parameter	Requirement	Actual Value
Dimensions	1.6×0.8 m ellipse	Same
Mass	10 kg	8.6 kg
Areal density	7.8 kg/m^2	6.7 kg/m^2
Stiffness (natural frequency)	80 Hz	228 Hz (calculated)
Surface accuracy (as-fabricated)	8.5 μm rms	4.37 μm rms
Surface stability (on-orbit environment)	18 μm rms	6.1 μm rms
Solar absorptance	0.40	0.43
Absorptance/emittance (α/ε)	$1 < \alpha/\varepsilon < 2$	1.3

A high accuracy coordinate measuring machine was used periodically during the fabrication process to measure the figure accuracy of the reflector. These measurements confirmed that the mirror surface directly replicates the mold surface within a factor of two. This data suggests that higher figure accuracies can be obtained through further refinement and polishing of the mold surface. Despite the small surface errors, the final figure accuracy value, 4.37 μm rms, is well within the 8.5-μm specification. This project demonstrated the feasibility of fabrication low-mass, high-figure accuracy reflectors using composite technology.

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CHAPTER 37

SELECTION OF MATERIALS FOR BIOMEDICAL APPLICATIONS

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1 INTRODUCTION

Materials have been used for medical implant applications for centuries. Starting even before George Washington's famous wooden teeth, before the use of coconut shells in the 1800s by South Seas natives to replace missing portions of the skull (Sanan and Haines, 1997), humans have attempted to use materials from biological and inorganic sources to replace diseased or damaged tissues. In fact, some examples of biomaterial implants, particularly in the form of gold and silver, date back to prehistoric times (Sanan and Haines, 1997). These material selections of the past, based more on the availability of materials and anecdotal evidence than scientific method, resulted in varying degrees of success. The development of aseptic surgical technique in the midnineteenth century by Lister provided the basic medical tool needed for the more widespread and successful use of biomedical implants (Park and Lakes, 1992). Since the twentieth century, as knowledge of the biological mechanisms behind the interactions of implanted materials and tissues has increased, the selection of materials for medical implants has been based on progressive improvements and experimental evidence.

When selecting a material for use in a medical implant application, two general considerations need to be taken into account: the functional requirements

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Handbook of Materials Selection. Edited by Myer Kutz

of the implant and how it will interact with the body. The function of the implant includes the physiologic role that it will replace, as well as the length of time that it is designed to fulfill that role. The interaction of the body and the implanted material must be examined from two perspectives—the effect of the biological environment on the material properties and the effect of the material, and any degradation that may occur, on the local and systemic physiology of the body.

It is not possible within the scope of this chapter to discuss the optimum choice of materials and the supporting rationale for every medical implant application. This task is made even more difficult by the fact that the development and selection of materials is an ever-changing field. In light of this fact, the following pages will use three implant examples to serve as the background for a discussion of the considerations in the selection and evaluation of materials for medical implants. Each section will first be organized around the functional requirements of the implant, with the resulting concerns regarding material–tissue interaction being discussed for the materials that would first meet the functional demands. Geometrical considerations and overall implant design will also play a major role in the success of an implant; however, this chapter will focus on the selection of biomaterials as a discrete step in the design process.

2 ORTHOPEDIC BIOMATERIALS: TOTAL HIP ARTHROPLASTY

The hip (Fig. 1) is one of the most commonly replaced joints, due to the high incidence of both osteoarthritis and osteoporotic hip fracture within the population. As with the other joints in the skeletal system, the hip has two main functions: (1) to transfer load from one bone to another and (2) to allow for motion between the bones. The loads on the hip have been found to vary from 0.5 to 8 times body weight during activities of daily living, including brisk walking (Paul, 1999). The loads can be expected to reach even higher levels during events such as a stumble. The hip is essentially a ball-and-socket joint

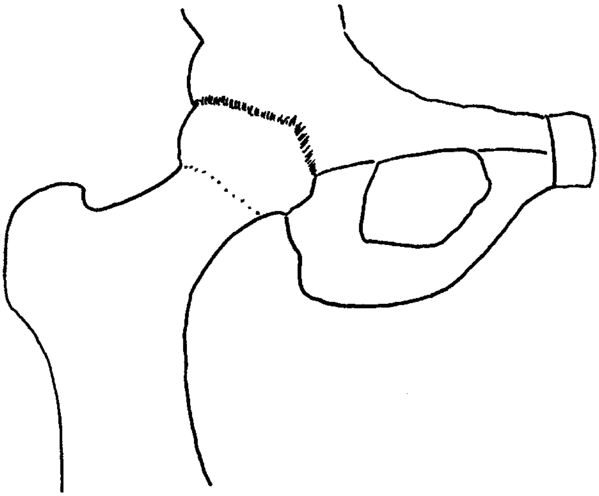

Fig. 1 Anatomy of the hip.

with 6 degrees of freedom constrained by the bony and ligamentous structures of the joint. These functional constraints have generally defined the goals for design of hip implants over the past half-century.

The total joint replacement for the hip is divided into three components: the femoral stem and femoral head, which may be integrated or modular, and the acetabular cup (Fig. 2). The combination of the femoral head and the acetabular cup provide the bearing surface for the joint. The femoral stem transfers the load to the femur and provides resistance to the bending moment caused by the anatomy of the joint. Total hip replacement began in the 1930s, with stainless steel used for both the acetabular cup and the femoral head, which was bolted onto the natural femoral neck. In the 1950s, the general design of the implant was expanded by McKee to include the stemmed femoral component familiar today. In 1959, Charnley introduced a plastic acetabular component, which articulated with the metallic femoral component. This idea for low-friction arthroplasty remains the basis for the predominant implant designs used today (Swanson, 1977).

2.1 Function

Load Support

Because of its primary role in mechanical support, the stem of a femoral prosthesis can realistically be manufactured from a metal, a ceramic, or a composite material. For this discussion, composite materials will not be included, as they have not yet been utilized in commercially available total joint replacements (they are available for bone plate applications). However, as development of

Fig. 2 Example of total hip implant, including femoral and acetabular components.

composite materials for this application continues, they must meet the same requirements to be discussed for ceramic and metallic materials.

Strength. The first property to be considered for a load-bearing implant is its mechanical strength. The loading of the femur is dynamic and, while estimated to range up to 8 times body weight, is difficult to determine precisely. Therefore, as the implant will be loaded in essentially the same way as the natural bone, it is reasonable to assume that a material that will provide the same or greater load-bearing capacity as bone will meet the necessary mechanical requirements. The mechanical properties of cortical bone (Table 1) form the lower limit for properties of materials to be selected for the femoral stem. If the neck of the femoral stem is designed to be longer than the normal range of femoral neck lengths [approximately 9 cm, from the edge of the greater trochanter to the apex of the femoral head (Center et al., 1998)], then the increased bending moment in this region should also be considered. The primary mode or modes of loading that will be seen for a particular implant application will also be important factors in the determination of whether a material meets the constraints regarding mechanical strength. The hip will be loaded primarily in bending and compression, due to its unique geometry. Tensile and shear strength of any replacement material therefore become of great importance.

In addition to the yield or ultimate strength of an implant material, the fatigue strength is also important for structures that will be cyclically loaded over an extended period of time. At the current time, hip replacements are designed to last for approximately 20 years. As an individual typically loads and unloads the joint thousands of times per day during normal daily activities, fatigue-induced failure at this location is of paramount concern. In contrast to the materials that might be chosen to replace the original tissue, healthy bone has the capacity to repair any microfractures that may occur through continuous cycling. This normal remodeling function helps to eliminate the occurrence of fatigue fractures in normal bone.

Based on the cyclic loading that a material is likely to undergo when implanted in the body, it is reasonable when evaluating selections to compare the endurance limit of the materials under consideration to the experimentally determined strength values for bone. One further complication in this process, however, is that materials will fatigue differently in a typical, air-based laboratory environment than they will in the ionic soup of the human body. Because of this, it is important to measure the fatigue performance of materials in an environment that closely mimics that of the implant location—including the ionic composition, temperature, and pH. Substantial work has been conducted

Table 1 Representative Properties of Cortical Bone

Compressive strength (MPa)	131–224 longitudinal 106–133 transverse
Tensile strength (MPa)	80–172 longitudinal 51–56 transverse
Shear strength (MPa)	53–70

Source: Data from Cowin (1989).

to develop testing methodologies for this purpose, and many have been compiled by the American Society for Testing and Materials (ASTM), the International Standards Organization (ISO), and other bodies interested in standardization. For instance ISO-7206 (1989) discusses many aspects of testing for partial and total hip replacements. Thus, individuals who are working to design new materials for biomedical implants do not need to reinvent the wheel when it comes to evaluation tests.

Finally, when the mechanical strength of a potential implant material is evaluated, the failure behavior of the material must be taken into consideration. The ability of the material to absorb energy during potential abnormal loading events, such as a fall or a jump, will affect its overall, long-term performance. Metals are significantly less brittle than ceramics, and as a result are less likely to fail during a high-energy or high-rate loading event. Ceramics are also more susceptible to failure in bending than metals, due to their relatively low tensile strength and low resistance to crack propagation. These factors do not eliminate ceramics from consideration for orthopedic implants; however, an understanding of a material's behavior under a range of loading modes and rates is needed before a final determination of its suitability for a particular application can be determined.

Stress Shielding. In bony prosthetics, exceeding the minimum strength requirements of the application is not the only mechanical behavior of concern. Bone is a living tissue and not merely a structural material. As a result, bone changes in response to its loading environment. In 1892, Wolff first noted that the pattern of the trabecular bone in the head and neck of the femur was similar to the stress trajectories of a Culmann crane, an engineering structure with a similar loading pattern (Wolff, 1892). Based on this observation, Wolff hypothesized that bone develops and remodels in response to the load that it experiences. Over 100 years later, Wolff's law is still the governing principle behind our understanding of how bone behaves in response to stress. In general terms, bone requires a minimal, time-averaged stress in order to maintain its mass. If the stress falls below this threshold level, bone will be lost. If the stress increases beyond this threshold, bone will be added until the stress experienced returns to its "desired" level. This is evidenced by the loss of bone seen in individuals after sustained bed rest or in astronauts after missions spent in low-gravity environments.

As a result of this characteristic property, bone is susceptible to a phenomenon termed stress shielding. This occurs when the stress in bone is reduced below its maintenance threshold as a result of the mechanical role of an implanted structure. Figure 3*a* shows the radiograph of a femur in which significant bone loss has occurred around the proximal end of the implant, with stress shielding being one of the underlying causes. Bone loss such as this is one of the leading reasons for implant failure and revision. The formation of a callus at the distal end of the implant, as also shown in Fig. 3*b*, is additional evidence of the response of bone to its stress environment. In many hip implant designs, while the stress in the bone in the proximal region of the implant is reduced, an increase in stress is seen in the distal region that results in the deposition of additional bone.

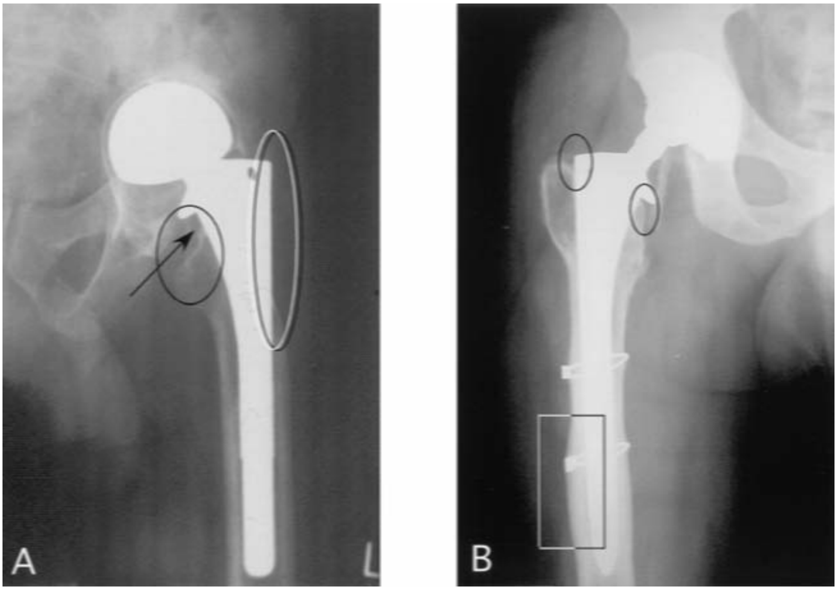

Fig. 3 X-rays of hip implants showing changes in bone mass resulting from stress shielding around an implant. (A) Circled areas indicate regions of significant bone loss in the proximal region of the femur. The arrow indicates the area where the collar of the prosthesis is no longer in contact with bone on the medial side. (B) Proximal bone loss (indicated in the circled regions at the edge of the prosthesis) is accompanied by increased bone deposition (indicated by the rectangle) in the distal region of the implant. The latter phenomenon is caused by increased stress transfer in this region.

In any loaded structure consisting of two or more materials, the distribution of stress and strain between the component materials will depend on their geometric arrangement and relative material properties. For a structure in which the materials are in parallel with the loading axis and where the materials are sufficiently well bonded—such as a well-fixed femoral implant—it can be assumed that the materials deform to the same extent and therefore experience the same strain. In this isostrain condition, the stress in one of the components of a two-phase composite can be calculated from the equation:

$$\sigma_1 = \frac{E_1 P_1}{E_1 A_1 + E_2 A_2} \tag{1}$$

where P is the total load on the structure, and E and A are the Young's modulus and cross-sectional area of each of the components. Thus, the fraction of the load carried by each material, and the resulting stress, is related to its Young's modulus and cross-sectional area in relation to those of the other components of the composite structure. The stiffer materials in the composite will carry a greater proportion of the load per unit cross-sectional area.

As stated above, the important parameter for maintenance of bone is the stress that it experiences compared to a threshold value. If bone in its natural state is compared to bone with an implant, the effect of this intervention on the stress in the bone, and therefore its remodeling response, can be estimated. For a femoral implant, or a bone plate as another example, the implant is in parallel with the bone tissue with respect to the main loading axis. Therefore, Eq. 1 can be used to estimate the effect of the implant on the stress in the bone. The applied load can be assumed to be the same pre- and postimplant, which yields the following equations for the stress in the bone in the two configurations:

Preimplant ($E_{\text{implant}} = 0$; $A_{\text{implant}} = 0$)

$$\sigma_{\text{bone}} = \frac{E_{\text{bone}} P}{E_{\text{bone}} A_{\text{bone}}} = \frac{P}{A_{\text{bone}}} \tag{2a}$$

Postimplant

$$\sigma_{\text{bone}} = \frac{E_{\text{bone}} P}{E_{\text{bone}} A_{\text{bone}} + E_{\text{implant}} A_{\text{implant}}} \tag{2b}$$

Thus, the stress in the bone is reduced by the inclusion of the implant, with the amount of the reduction dependent on the modulus and area of the implant. Implants with a higher modulus and a larger cross-sectional area will shield the bone from a greater proportion of its normal, physiological stress, resulting in bone loss according to Wolff’s law.

Joint Motion

Friction. Frictional forces between the articulating surfaces of a joint have two primary effects: (1) to increase the muscle force required to overcome the internal friction and allow motion to occur and (2) to increase the torque experienced by the implant and/or bone, such as at the location of the femoral neck. Large internal bending moments due to high frictional forces may lead to failure of the implant, and therefore should be avoided.

The natural joint, with its cartilage bearing surfaces and synovial fluid lubrication, possesses a remarkably low coefficient of friction that minimizes the tangential and bending forces at the joint. It has been recognized that, in an artificial hip, the coefficient of friction between the femoral head and the acetabular cup must be minimized in order to most closely approximate the normal physiology. While no material combinations currently provide a coefficient of friction of the level seen naturally, a number of options have been identified that provide sufficiently low friction forces in the joint. Table 2 provides the coefficients of friction of some commonly paired materials used in hip replacement in comparison to the natural state. Once implanted, the joint will be lubricated with physiologic or synovial fluid, although it will no longer be contained within the original joint capsule. Therefore, the friction values provided were measured using physiological fluid or bovine albumin as a lubricating material. Alumina has a relatively high surface tension, allowing it to develop a good lubrication film that minimizes friction *in vivo* (Ravaglioli and Krajewsk, 1992).

As friction forces are proportional to the coefficient of friction and the normal force between the contact surfaces, the loading configuration of an implant will also determine the forces that influence joint motion and applied bending mo-

Table 2 Coefficient of Friction for Sample Material Combinations Used in Total Hip Replacement[a]

Material Combination	Coefficient of Friction
Cartilage/cartilage	0.002
CoCr/UHMWPE	0.094
Zirconia/UHMWPE	0.09–0.11
Alumina/UHMWPE	0.08–0.12
CoCr/CoCr	0.12
Alumina/alumina	0.05–0.1

[a] UHMWPE, ultra-high-molecular-weight polyethylene; CoCr, cobalt–chromium alloy.

Source: Data from Park and Lakes (1992), Streicher et al. (1992).

ments. Therefore, materials that will provide a sufficiently reduced friction environment for a relatively low force location, such as the wrist, can result in a friction force in the hip that may cause implant failure to occur or cannot be adequately overcome by normal muscle forces to allow unhindered motion. Thus, once again, the specific application of an implant must be considered when selecting materials—generalized assumptions and choices do not necessarily produce the optimum selection.

Wear. Whenever contact surfaces and motion are combined, material wear must be taken into consideration. Wear is the process whereby one object, through motion, removes material from the surface of the contacting object. Generally, the harder material will cause wear to occur on the softer material. Three basic types of wear can occur: abrasive wear, adhesive wear, and third-body wear. Abrasive wear exists when a hard material, such as a metal, moves cyclically against a soft material, such as a polymer. Adhesive wear involves the sliding motion of two similar materials, where molecular bonds can be formed at the interface of the structures. In rough materials, the surfaces appear as a series of peaks and valleys. The two articulating surfaces typically come into contact at the peaks of the surface roughness, concentrating the contact load over a much smaller area and increasing the contact stress. As the molecular bonds between the objects are broken through motion, they also break off particles of the underlying material. Third-body wear includes the effect of particles between the articulating surfaces that tend to accelerate wear.

The amount of wear that occurs between two surfaces will depend on several factors: (1) the hardness of the two materials (p), (2) the normal force at the surface (F_n), (3) the Archard coefficient for the pair of materials (k), (4) the area of contact between the surfaces during the cyclic motion, and (5) the number of cycles expected. Archard's coefficient is similar in concept to the coefficient of friction and describes the degree to which a normal force at the surface is translated into a wear-producing force. A harder material will always sustain less wear when in motion against a given surface than a softer material. As two materials sweep out a larger surface of contact for each cycle of motion, there will be a greater fraction of the surface area experiencing wear, thus resulting in an increase in the overall rate of wear. And, with little explanation required, if a given amount of wear occurs per cycle, then an increased number of cycles will result in a greater total amount of wear.

The wear rate, or volume of wear particles produced (V), can be approximated for adhesive wear by the equation:

$$V = \frac{kF_n x}{3p} \tag{3}$$

where x is the total sliding distance between the surfaces (Black, 1999). The total sliding distance can be determined for a single cycle, a defined period of time (e.g., one hour), or estimated for the entire life of the implant, thus allowing for the calculation of a rate for wear particle production.

Wear can be exacerbated through corrosion or fatigue processes, indicating that *in vivo* wear rates may differ from those measured in laboratory. The occurrence of wear in an implant may affect both its mechanical function and the response of the body to the implant. The former will be discussed here, while the effect of wear debris will be described in the discussion of biocompatibility.

As wear occurs on one or both of the opposing surfaces of an articulating joint, the shape of the implant may change as material is removed. This is especially evident when significant wear occurs in a polymeric acetabular cup within a total hip replacement. Figure 4 shows how significant wear can result in the creation of a new socket, typically with a reduced diameter. This change in the configuration of the acetabulum can affect the range of motion of the hip as well as whether any joint impingement occurs. As the socket deepens, the femur will shift upward, which will increase the laxity of some of the ligaments and tendons, thus affecting overall joint performance.

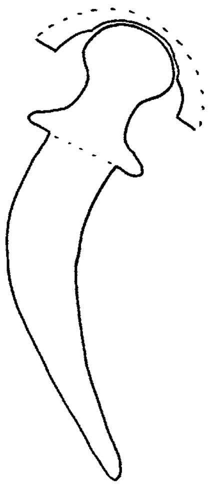

Fig. 4 Illustration of the development of a new socket within an acetabular cup due to excessive wear.

2.2 Biocompatibility

Once a material is selected for an implant application based on the functional requirements, it must be evaluated in terms of material–body interactions.

An implant material will react chemically with the local environment, with the type of reaction dependent on the class of material. Metals are susceptible to corrosion, polymers experience leaching and absorption, while ceramics are generally considered to be chemically inert—unless designed to be bioactive. The effects of chemical degradation may affect both the tissue and the material itself, especially its mechanical properties, and so both aspects must be considered. In addition, degradation products can affect the physiology locally, at a remote location, or systemically.

Corrosion

Metallic materials are susceptible to corrosion, particularly in the ionic fluid environment of the body. To assess the corrosion potential of a metal, it is necessary to examine the half-cell potential of that metal—which will act as an anode when it releases electrons—with respect to the material acting as the cathode. This cathode may be another metal or the ionic environment itself. An electrochemical series lists the half-cell potentials of metals in order from the most noble (or cathodic) to the most anodic. When two materials are in contact with each other directly or through an ionic solution, the metal listed first in the list will act as the cathode while the other will behave as the anode. Practical electrochemical series typically relate half-cell potentials as measured in an application-specific environment and may include alloys. This contrasts with ideal series, which list only pure metals as measured with respect to a hydrogen half cell reaction. The ideal series approximates the behavior of metals in pure water. Table 3 shows the half-cell potentials of common metals as measured in an ideal

Table 3 Ideal and Practical Electrochemical Series for Common Metals[a]

Ideal Series	Half-Cell Potential (mV) of Ideal Series with Respect to H/H+ Half-cell	Practical Series
	Cathodic	
Gold	−1.50	Platinum
Platinum	−0.86	Gold
Silver	−0.80	Passivated stainless steel
Copper	−0.47	Titanium
Lead	0.12	Silver
Tin	0.14	Unpassivated stainless steel
Nickel	0.23	Copper
Iron	0.44	Tin
Chromium	0.56	Lead
Aluminum	1.70	Wrought iron
Titanium	2.00	Aluminum
Magnesium	2.40	Magnesium
	Anodic	

[a] The half-cell potentials listed are for the ideal series and are with respect to a H/H^+ half-cell.

Source: Data from Black (1999), Park and Lakes (1992).

electrochemical series, as well as the qualitative series of metals in a saltwater solution. This latter series reasonably approximates what would be found in the body, where sodium chloride is a major constituent of the ionic soup.

The low half-cell potentials of gold and platinum are a result of their essential inertness. For other materials, their resistance to corrosion may be due to the formation of a protective oxide layer on the object's surface. A second way to determine how a metallic material is likely to behave *in vivo* from a chemical standpoint is to examine Pourbaix diagrams that have been developed based on theoretical chemical relationships. These curves, such as the one shown in Fig. 5, describe the expected corrosive behavior of materials as a function of pH and the surrounding electrical potential. By determining the expected environment at an implant site, it is possible to predict which materials are likely to meet the outlined requirements for implantation. In regions of buffered pH, which are common within the human body, the partial pressures of O_2 and H_2 can be used to estimate the electrical potential at the site (Black, 1999).

Three general behaviors can exist for a metallic material in an ionic environment: corrosion, passivation, or immunity. Corrosion is the chemical reaction in which a metal is oxidized, producing metallic ions within the fluid environment. As a metal corrodes, it can effect both the overall implant properties—resulting in premature failure—and the body. In the latter case, the ions produced through the oxidation reaction can interfere with the normal physiologic process of the body at either a local or a systemic level. It cannot be assumed that the ions produced will have no adverse reaction in the body if they are normally present in trace amounts. For instance, iron, a mineral required for normal red blood cell production, will prove to be toxic at elevated concentrations in the body, causing liver and pancreatic failure (Smith, 1983). Therefore, it is important to select materials that will exist within their passivation or immunity regions in the *in vivo* environment. Passivation is the creation of a protective, oxide or

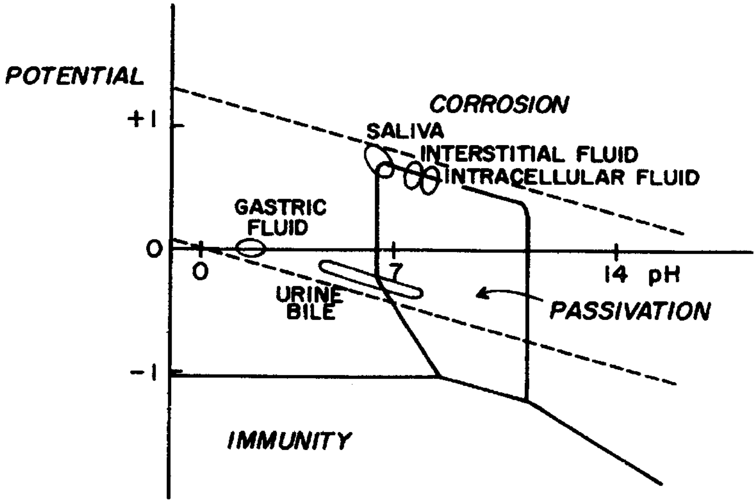

Fig. 5 Pourbaix diagram for chromium in aqueous solution of 1 N Cl^-. The diagram indicates the electrochemical environment of various regions of the body. [From John H. Dumbleton and Jonathan Black, *An Introduction to Orthopaedic Materials,* Thomas Publishing, Springfield, IL, 1975, with permission].

hydroxide layer on the surface of the metal. Immunity is the region in which minimum oxidation is going to occur as the equilibrium concentration of metallic ions is low. (The equilibrium concentration refers to the levels of ions in the immediate, surrounding environment at which the oxidation reaction reaches equlbirium, thus producing no further net release of ions.) Both passivation and immunity require that less than 10^{-6} M of ions be produced (Park and Lakes, 1992). Thus, in neither of these cases is oxidation eliminated for a metallic material; however, the effect on the implant's properties and the ion concentration produced will be negligible. As a final check of biocompatibility, it is important to ascertain that the trace levels of ions produced by passivated or immune metals will not produce toxic effects at even those low concentrations.

Pourbaix diagrams can only provide information on the ideal behavior of a metal within its immunity or passivation regions. As the limitation of corrosion in these environments is due to the establishment of a chemical equilibrium, the ionic environment of the implant surface and the surrounding physiologic fluid must remain undisturbed. Therefore, any damage to the passivation layer, or any fluid flow that removes the equilibrium ions from the region immediately adjacent to the implant, may cause renewed corrosion. Cyclically loaded implants could be expected to experience both surface damage, due to wear or contact with other tissues, and movement of the fluid surrounding the implant. In addition, the local pH at a site may be affected by common physiologic insults, such as injury and infection. Finally, most Pourbaix diagrams are determined for pure water environments, and therefore do not include the role of additional ionic components in the physiologic fluid. This would also be expected to influence the corrosion behavior of a material. Therefore, the behavior predicted by the Pourbaix diagrams may be taken as a base line expectation, but must be confirmed through both animal and clinical testing.

Some additional rules can be applied to minimize the occurrence of corrosion for an implant with metallic components. These are:

1. *Minimize the number of different metals used in the implant.* Two metals that differ in electrochemical potential, due to differences in elemental composition or processing, will create a galvanic potential when connected either directly or through an ionic fluid. This can include parts manufactured of the same material but by different companies or different techniques, as the processing schedule may differ enough to create differences in the electrical potentials of the components.
2. *Minimize the possibility of surface damage.* Any scratches to the surface of a metallic implant can serve as foci for corrosion once it is implanted. Thus, it is important to protect the implant from the stage of final processing through implantation to prevent damage.
3. *Reduce the number of crevices or regions where oxygen depletion is possible.* A region with a low-oxygen concentration, such as the interfaces between modular components of a total joint implant or the threads of a bone screw, is likely to become anodic with respect to the surrounding metal—even with respect to the remaining bulk of the metal. Corrosion will therefore occur with the small, anodic region providing electrons to

the entire cathode. If there is a discrepancy in the size of the regions, as would normally be expected, then corrosion within the crevice can be accelerated, leading to potential mechanical failure.

Leaching and Absorption

Polymers placed in a fluid environment can experience two opposite phenomena. In leaching, unreacted monomer molecules, fillers, or small chains of polymers can diffuse from the bulk of the polymer to the surrounding fluid. As in corrosion products, these released molecules may have a negative effect on the local physiology or, if transported through the bloodstream or lymphatic system, on systemic or remote processes. In addition, significant leaching may reduce the density of the polymer and consequently have an adverse effect on the properties of the structure. Absorption occurs when water molecules, proteins, or lipids diffuse from the fluid into the mass of the polymer. The absorbed molecules become distributed between the molecules of the polymer, reducing the mechanical strength of the structure and increasing its susceptibility to wear.

Both absorption and leaching occur as a result of diffusion processes across the surface of the implant. Fick's first law of diffusion describes the rate of solute transfer across a permeable barrier:

$$F = -D\left(\frac{\partial C}{\partial x}\right) \tag{4}$$

where F is the rate of solute transfer per unit cross-sectional area of the surface, D is the diffusion coefficient, and $\partial C/\partial x$ is the concentration gradient. The diffusion coefficient is dependent on the solute (the molecule that is being absorbed or released), the matrix through which it is moving (the remainder of the bulk polymer), and the type of diffusion that is occurring. Thus larger molecules moving through more tightly bonded matrices are likely to leach out at a lower rate than small molecules diffusing through a more open, amorphous structure.

All materials, including metals and ceramics, can absorb molecules—particularly water—from the surrounding environment. However, this occurs much more readily in the relatively loosely bonded polymers. Absorption in polymers can also result in swelling, due to their low elastic modulus, which may cause geometric changes that interfere with the performance of an implant. The strain that a polymeric object experiences due to swelling may induce cracks and may also reduce the ultimate strength of the object. This latter phenomenon occurs because, due to the new baseline strain in the material, less stress is needed to reach the material's ultimate strain. If the absorbed molecules are small, such as water, they will act as plasticizers and weaken the bonds between the polymer chains, thus reducing the Young modulus of the material. If a polymer is hydrophobic in nature, it is less likely to absorb water. However, absorption of nonpolar molecules such as lipids may still occur.

Leaching generally has a smaller effect on the mechanical properties of a polymeric structure than absorption. However, as was the case in corrosion, local changes in properties may occur as material is lost. In fact, if sufficient leaching occurs to create adjacent or expanded voids within the chemical structure of the polymer, these regions may act as stress risers. A high amount of leaching will act to increase the porosity of the polymer. In both of these cases, the elastic modulus and the mechanical strength of the polymer would be affected.

In general, low-molecular-weight or highly amorphous polymers are more susceptible to leaching or absorption processes than high-molecular-weight, highly crystalline, or highly cross-linked polymers. As diffusion coefficients increase for smaller molecule sizes, in general, it can be expected that additives and free monomers have a higher rate of leaching than the large polymer chains themselves, while the relatively small water molecule will be absorbed by hydrophilic polymeric structures at a relatively high rate. The behavior of free monomers and additives within polymers is of particular interest, as they may have a different effect on the surrounding tissue than tests based on the bulk polymer suggest. In fact, free monomer should be expected to have a different physiologic effect than molecules that have reacted to form the bulk polymer, due in part to the difference in chemical reactivity of the structures.

Wear Debris

The material produced through the wear process is typically in the form of particulate debris. The size of the particles is dependent on the material involved and may range from submicon dimensions to millimeter-sized pieces. In polyethylene, particles typically range from 0.5 to 50 μm in their largest dimension, while polymethylmethacrylate (PMMA, bone cement) particles tend to be significantly larger (Willert and Semlitsch, 1996). These particles can have several adverse effects on implant performance beyond the geometric changes discussed above. First, if the debris becomes trapped between the articulating surfaces of the joint, it will act as a collection of third-body particles to accelerate the wear process. Second, the presence of particulate debris in the tissue surrounding the bone triggers an immune response that can result in significant bone loss through the process of osteolysis. When this occurs without an associated infection, it is termed aseptic loosening. The presence of histiocytes, macrophages, and foreign body giant cells in locations with both mild and severe osteolysis indicate that the process is inflammatory or immunological in nature. It is currently hypothesized that the presence of particulate debris results in macrophage and giant-cell recruitment (Jacobs, et al., 1994). The larger particles are engulfed by giant cells through phagocytosis and form granulation tissue, while macrophages react with the smaller particles. Normally, foreign debris on a small scale will be removed through the lymphatic system. However, if the volume of debris produced overloads the lymphatic system, then macrophages at the implant site may release cellular mediators that trigger the bone resorption observed.

2.3 Current Material Selection

The traditional materials that have been used for total hip arthroplasties are ultra-high molecular-weight polyethylene (UHMWPE) for the articulating surface of the acetabular cup and a metal, today typically an alloy of titanium or of cobalt–chromium, for the femoral stem, head, and backing of the acetabluar cup. In a smaller number of designs, ceramics such as alumina (Al_2O_3) and zirconia (ZrO_2) have been used for a modular femoral head component, both to reduce friction within the acetabulum and minimize the number of metallic components that may exacerbate corrosion. Table 4 provides a summary of some of the

Table 4 Summary of Mechanical Properties for Materials Commonly Used in Current Total Joint Replacement Designs[a]

Property	HMWPE	Titanium Alloy	Co–Cr Alloy	Alumina	Cortical Bone
Elastic Modulus [GPa]	2.2	110	220–234	350–400	10–20
Comp. Strength [MPa]				4000	130–280
Tensile Strength [MPa]	3	860	600–1000	270	80–160
Endurance Limit [MPa] at 10^7 cycles		620	500		
Density [g/cm^3]	0.93–0.94	4.5	9.2	3.9	1.8
Hardness [MPa]		3500	3000–4000	20,000	

[a] Titanium alloy, Ti6Al4V; Co–Cr alloy, wrought CoNiCrMo.

Source: Data from ASTM-F136 (1998), ASTM-F562 (2000), ASTM-F648 (2000), Bernache-Assolant (1991), Brunski (1996), Cowin (1989), and Park and Lakes (1992)

mechanical properties of these common materials and lists cortical bone for comparison purposes.

In the selection of metals, cobalt–chromium alloys with molybdenum have been preferentially chosen over those with nickel. Despite the fact that the mechanical properties are slightly reduced in the molybdenum alloy compared to the nickel variant, it has been shown to have improved wear properties. Titanium is often chosen due to the fact that its elastic modulus is half of that seen in cobalt–chromium or stainless steel alloys, therefore reducing the stiffness of the implant and the accompanying bone loss due to stress shielding.

During the past decade, substantial research has been conducted to develop different pairs of materials for the bearing surface in order to reduce the wear phenomenon commonly seen in HMWPE, which often leads to premature failure of an implant. Metal-on-metal and ceramic-on-ceramic head–cup combinations have shown promise in terms of long-term outcomes, especially when used in younger patients for whom an implant life of greater than 20 years would be desirable (Delaunay, 2000; Skinner, 1999; Wagner and Wagner, 2000). The ceramics typically selected have been alumina and zirconia, while the metal is generally a cobalt–chromium alloy. Titanium does not lend itself to metal–metal bearing surfaces due to the fact that it tends to seize when in contact with other metals. The success of these material combinations is due in large part to the improvements in materials processing, machining, and polishing that have been achieved in the last quarter century, allowing for the development of high-quality materials, excellent geometric matches, and highly polished surfaces. This progress also indicates that designs that were tried out at an earlier point in history, and were perhaps abandoned, may deserve to be reexamined. Metal-on-metal bearings were originally used in total hip replacements in the 1950s, with limited success. Complete ceramic bearings were seen as early as the 1970s, but fracture of the ceramic components and high wear were attributed to problems in processing that resulted in defects and inadequate grain sizes (Plenk, et al., 1992; Sedel, et al., 1991). In time, they were almost completely replaced in the clinical world by metal–polymer combinations. However, their revival in the past few years, spurred on by developments in materials engineering, indicates that the original developers had the right idea—they were just a few years ahead of their time.

3 BLOOD-CONTACTING BIOMATERIALS: VASCULAR PROSTHESES

When blood vessels are damaged through injury or disease, they often must be replaced or bypassed in order to maintain adequate blood flow to and from the regions of the body. Disease-induced damage, such as atherosclerosis and aneurisms, occurs more often in arteries than in veins, due in large part to the higher working pressure of the blood within these vessels. Injury can occur to any blood vessel; however, collateral circulation typically eliminates the need to replace small veins, and the low venous return pressure provides an environment in the larger veins that is much more conducive to traditional surgical repair or autograft use. As a result, this section will focus on the selection of materials for the development of arterial prostheses.

Arteries are three-layer hollow tubes (Fig. 6) composed of a combination of elastin, collagen, and smooth muscle—with the proportions of each component dependent on the size of the artery. The size of an artery also varies substantially along the arterial tree, from a typical diameter of about 4 cm for the aorta in an adult to a diameter of less than 1 mm for arterioles. The age and size of the individual will also affect the dimensions of the arteries. Normal arterial pressure is approximately 120 mmHg during the systolic phase (contraction) of the cardiac cycle, decreasing to approximately 70 mmHg during the diastolic phase (relaxation). However, pressures can rise as high as 200 mmHg or more in individuals with either transient or chronic hypertension, and hypertension itself is a risk factor for a number of the pathological processes that may require arterial replacement or bypass.

Elastic arteries, such as the aorta, are expected to expand during systole as blood is forced into them by the heart. The elastic recoil of these vessels, due in large part to the high amount of elastin in the wall, acts to maintain arterial pressure during the relaxation phase of the cardiac cycle, further transporting blood away from the heart. Distributing arteries, which (as their name indicates) distribute blood from the larger, elastic arteries to the various regions of the body, have more collagen than elastin in their walls. However, the physiologically important component of these vessel walls is the smooth muscle, which

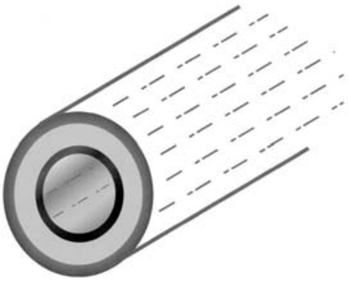

Fig. 6 Schematic diagram of an artery showing the three tissue layers: adventitia (outer), media (middle), and intima (inner).

allows the distributing arteries to adjust their diameter and therefore their resistance. By controlling the arterial resistance to blood flow in various regions of the body through vasodilation and constriction, blood can be directed to regions that have the highest metabolic demand—the muscles during exercise or the gastrointestinal system during digestion.

Vascular grafts were first introduced in the early twentieth century as solid tubular structures. Fabric grafts were developed in the 1950s and provide the basis for the grafts that are currently in use or under development. An arterial graft or arterial prosthesis has one obvious function—to transport blood from vessels that are proximal to the graft to those that are distal to the graft. In addition, the vasodilation/constriction and elastic recoil capabilities of the various types of arteries assist the heart with transport and direction of blood to the diverse tissues of the body. However, there are additional functional constraints that are equally as important. First, the prosthesis must maintain hemostasis—not allow blood to leak from the artery into the surrounding tissues. Second, interaction of the blood cells with the vessel must not act to initiate coagulation, which could result in thrombus formation and embolisms, nor hemolysis, the destruction of red blood cells. These functional constraints will be considered in the discussion on biocompatibility.

3.1 Function

Blood Transport

To transport blood between two connecting vascular segments, a tubular structure is required. However, given modern material processing techniques, this requirement does not itself limit the selection of materials substantially. The graft must also, however, be connected to the ends of the remaining vascular segments in some way, either through ligatures or sutures.

Using a maximum arterial pressure of 200 mmHg (30 kPa), the circumferential and axial wall stresses within a graft (diameter of 1 cm, thickness of 0.5 mm) can be approximated to reach 270 and 135 kPa, respectively. This is well within the mechanical limits of most undegraded artificial materials; however, it may become a substantial constraint for vessels engineered from natural materials in the future. In addition, the physiological environment, combined with the cyclic loading seen by the vessel due to the normal vascular pressure variations, will affect the properties of the graft material. As discussed above for hip replacement, corrosion, absorption, and leaching processes that occur when a material is placed *in vivo* will all negatively affect the ultimate strength of a material.

The expansion and recoil observed in elastic arteries must be taken into consideration when selecting a material for grafts at these locations. It is reasonable to assume that a relatively short length of the graft does not itself need to dynamically change dimensions to maintain blood flow through the segment of vasculature. However, any mismatch in the behavior of the graft to the connected vessels will result in substantial stresses on the ligatures, sutures, or the vessel itself. In certain applications, the graft may replace a substantial portion of an artery, such as in the descending aorta. In this case, elastic recoil in the graft will play an important role in maintaining the velocity of blood flow through the distal vessels, and the pulsatile flow may be important to maintaining function in organs supplied by the arterial tree (Mergerman and Abbott, 1983). The same philosophy would apply to distributing arteries. At the current time, no artificial material exists that would respond to the physiologic control mechanisms in order to dilate and constrict in conjunction with surrounding vessels. However, a graft that is less compliant than the attached vessel would result in substantial stress concentrations at the junction between the artificial and natural materials. This elevation in stress could lead to failure of the sutures or ligatures connecting the structure, as well as accelerate fatigue processes within the graft ends (Wilkerson and Zalina, 1994). In addition, a less compliant graft can also result in a stenosis in the vascular tree during systole, when the proximal segment of artery expands under pressure, but the blood then encounters the length of reduced diameter graft that acts to retard flow (Herring, 1983). These issues, along with the fact that blood vessels experience significant deformation due to general body motion, essentially dictate that a soft, flexible material be used for the graft.

An ideal graft would exactly match the compliance of the attached blood vessel, allowing it to expand and recoil to the same extent as the natural structure. This would require a nonlinear response, with an initial region of highly compliant stretch, attributed to the unkinking and realigning of the collagen fibres and a simultaneous stretching of the elastin. After the collagen in the vessel wall is straightened, its higher elastic modulus dominates the behavior of the vessel, and the compliance is significantly reduced. It is this behavior (Fig. 7) that is thought to limit the overexpansion of arteries during acute episodes of elevated blood pressure. The compliance of each segment of artery therefore depends on the proportion of collagen, elastin, and smooth muscle (which has a negligible effect on compliance in its passive state) within the vessel wall.

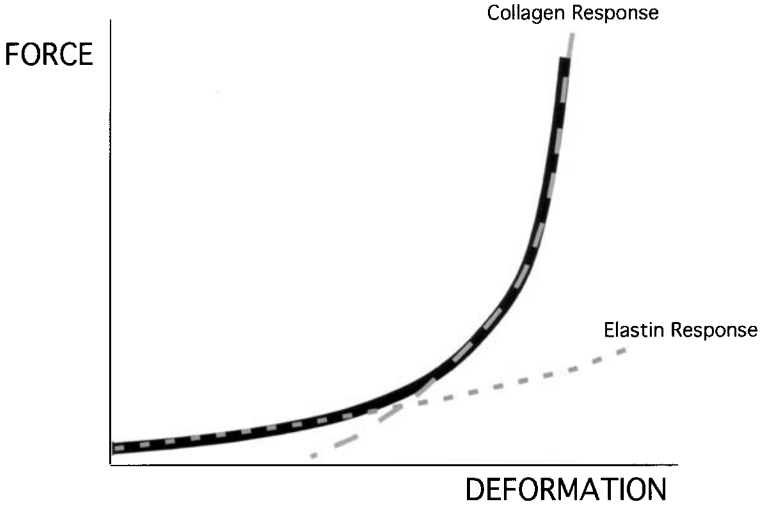

Fig. 7 Schematic diagram showing the characteristic nonlinear force–deformation relationship of an artery. Initial region of high compliance is characteristic of the elastin component in the walls, while the later, low-compliance region exemplifies the deformation of the collagen fibers.

Typical values for compliance (in units of percent radial change per millimeter of mercury) for cadaveric human arteries range between 10.9 at 60 mmHg and 3.8 at 150 mmHg (Mergerman and Abbott, 1983).

3.2 Biocompatibility

The primary functional need of a blood vessel—transport of blood—can easily be met through general implant design. However, due to the delicate nature of blood cells and the ease at which the clotting cascade can be initiated, biocompatibility issues place substantial limitations on material selection for this application. The natural vessel provides an optimal environment for blood flow, and the mimicking or replacing of its intimal layer is one of the underlying ideas in work to improve biocompatibility in vascular grafts.

Neointima Formation

Natural blood vessels consist of three layers: the outer adventicia, the middle media, and the inner intima. The intima has a layer of endothelial cells that contact the moving blood in order to minimize damage to the cellular elements of the fluid. Some artificial materials that are implanted into the body as vascular grafts have been shown to develop a *neointima*—fibrin and fibroblasts that develop as a lining on the inside surface of the tube (Herring, 1983).

The rate of neointima formation and its constituents may depend on the material used in the vascular graft. A carbon–ceramic composite with a large degree of surface porosity showed almost instantaneous (<10 s) development of a fibrin mesh with limited platelet adhesion (Chignier et al., 1987). This developed into a 5- to 7-cell thick layer of fibroblasts, collagen, and elastin by 15 days, with endothelial-like cells present and fully developed by 2 months. Herring (1983) provides a good description of the “healing” of vascular grafts and the development of the neointima. In most human vessels, complete endothelialization occurs only in the vicinity of the anastomoses with the natural vessels, as cells grow in from the ends of the connected tissue. To expand the area of vessel that experiences complete healing—such that the lining resembles that of a natural, healthy vessel—research is being conducted into seeding the vessel wall with endothelial cells (Consigny, 2000; Herring, 1983). This can either be done during the preclotting process, in the operating room, using a small number of cells taken from a vein that is exposed during the surgical procedure, or preoperatively using cells from the jugular vein that are cultured to provide a greater volume for the seeding process. In both cases, the use of autologous (patient specific) cells is important to eliminate issues of rejection.

The geometry of the vessel also affects neointima formation. The initial interaction between the blood and the graft is the formation of a clot, or thrombus, on the inner surface of the vessel. The thickness of the implant wall is directly proportional to the thickness of the thrombus formed on its surface (Park and Lakes, 1992). As the thrombus must be remodeled to form the mature neointima, a smaller clot results in faster organization of the neointima. Neointima formation must be limited, however, to prevent vessel occlusion. This is one of the issues with small diameter vascular grafts, where the fibrin layer may continue to grow past 1 mm in thickness (Wilkerson and Zalina, 1994). Finally, it is important to ensure that the neointimal layer that develops maintains its integrity and does not change in such a way, either through mineralization or adsorption of additional organic components, that it no longer promotes the continued patency of the vessel (Hufnagel, 1983).

Hemostasis

For vessels to adequately transport blood, the blood must remain within the lumen of the vessel and not leak into the surrounding tissues in any great amount. The obvious answer to this problem is to employ solid materials for vascular grafts, nonporous structures that will adequately contain the blood. However, the development of a neointima within a vessel provides a second design option, as it will act to seal a porous blood vessel against blood leakage. However, the time course for neointima formation is not short enough to provide the immediate seal needed during surgery and postoperatively. Therefore, many vascular grafts are preclotted in the operating room before they are implanted by exposing both the inner and outer surfaces to the patient's blood. This provides an initial surface that serves as the basis for neointima formation.

Hemolysis

Hemolysis, or the damage and destruction of red blood cells, occurs continually within the body. The cells are constantly replaced by new erythrocytes produced by the bone marrow. However, chronic damage to these cells and the release of their cellular contents into the plasma of the bloodstream can result in anemia, kidney failure, and other toxic reactions (Hershko, et al., 1998). Hemolysis due to nonphysiologic mechanisms typically occurs due to high shear stresses. A shear stress as low as 400 Pa can damage or rupture a red blood cell that comes in contact with another solid surface (Sallam and Hwang, 1984). Turbulent flow can also result in sufficient shear stresses to cause hemolysis. Therefore, two goals of vascular graft design are to minimize the contact between red blood cells and the graft surfaces and minimize the turbulence that may occur at branches or divisions of blood vessels. The latter falls within the domain of fluid mechanics, rather than biomaterial selection.

To minimize the contact between blood cells and graft walls that may lead to hemolysis, it is desirable to form a natural tissue layer between the two. Graft materials with high surface tensions tend to initially attract platelets and fibrin molecules, which aggregate and form the natural boundary that is then remodeled to become the neointima. The elements of the tissue layer also exhibit a slight negative electrical charge (Collins, 1983), which may act to repel the negatively charged red blood cells, therefore additionally reducing the contact between the cells and the vessel wall.

Coagulation

Blood clotting, or coagulation, is a necessary physiologic process that allows the body to heal and maintain its blood pressure through hemostasis. However, the formation of stationary clots within blood vessels (thromboses) and the movement of those clots with the blood flow (emboli) can result in vascular blockage, tissue damage, and even death. Therefore, it is important to select materials for graft use that will minimize the initiation of the clotting cascade.

The effect of a material on blood clotting is not easy to assess, particularly as it is difficult to separate the effect of the material from the natural physiologic process of coagulation. Blood will also behave slightly differently *in vivo* than it will *in vitro* and, like other tissues, can differ between individuals. Therefore, an *in vitro* coagulation test with blood from a dog may not be fully indicative of how that material will behave when implanted into a human. Standards have been developed, however, for coagulation tests (Bruck, 1980), and these progress from static, *in vitro* tests to dynamic, *in vivo* tests.

Surface tension, surface charge, and surface roughness are properties of a material that will affect the rate and amount of coagulation that takes place when it is in contact with blood. Polymeric materials tend to adsorb a mixture of proteins to their surfaces in the first 30–60 s of contact with physiologic fluid (Baier, 1975), the composition of which depends on the polar or nonpolar nature of the polymer (Herring, 1983). Adsorbtion and activation of key molecules from blood, including Hageman factor, factor XI, and others, will trigger the clotting cascade (Forbes, 1993). Rougher surfaces, including crimped grafts, have been shown to increase the rate of coagulation that occurs when in contact with blood (Collins, 1983). This is probably due to the larger surface area that can come in contact with the blood. A rough surface may be desirable, however, in order to promote preclotting on the surface of a porous graft. In the case of crimping, the process also prevents kinking of the vessels during surgery or prolonged implantation, which itself can lead to occlusion. Surface charge can help to minimize the contact between the graft and the blood elements. The formed elements of the blood—red and white blood cells and platelets—have been shown to have a negative surface charge. As a result, a vascular surface with a slight negative charge, through the presence of either a neointima or a naturally or artificially induced surface charge, will act to repel the blood cells and platelets away from the vascular wall (Collins, 1983). When platelets do not come in contact with the vascular wall or foreign bodies, they are less prone to initiate the clotting cascade.

The natural lining of blood vessels possesses unique properties that cannot be easily mimicked. It was originally believed that the smooth surface and the negative surface charge were the properties of an endothelial lining that prevented clot formation. However, studies have shown this to be more complex. In particular, the presence of endothelial cells allows for the secretion of paracrine agents that act to break down small thromboses and interfere with clot formation (Bruck, 1980). Smooth muscle and fibroblasts did not exhibit the same function, and in some cases precipitated platelet activation. Thus, as was the case in bone's resistance to fatigue, the living function of vascular tissue cannot be fully replaced using current technologies and artificial materials.

To reduce clotting in artificial materials, several approaches have been taken. Heparin, a negatively charged polysaccharide that is commonly used to prevent clottting in many clinical applications, has been coated on implants. Using the same logic, anionic radicals have been included in an artificial material to produce the negative surface charge that only naturally can occur in polymers. Taking a different approach, materials with low surface tensions have been proposed, as they are less likely to attract the formed blood elements to the material surface and initiate a clotting cascade. This last tact is, of course, in contradiction to the suggested use of high surface tension materials to minimize hemolysis!

This latest dilemma is indicative of many decisions that must be made in selecting a material for use in biomedical applications. No material—besides possibly the original tissue—will perfectly match all of the goals and constraints of the design. It is generally necessary to weigh the benefits and drawbacks of each material that makes the short list and to select the one that shows the best balance. While this is frustrating from a design aspect, it is what spurs material development and allows for the continuing work in the area of material synthesis and implant design.

Property Degradation

Once a material is selected that does not seem to have a significant, adverse effect on the blood itself, it is necessary to evaluate how the properties of the graft are affected during the expected contact with the physiological environment. As discussed above for orthopedic implants, polymers can react to the physiologic environment by either leaching smaller molecules into the surrounding tissue or absorbing water or other materials from the tissue. In the case of vascular grafts, the constant flow of blood past the implant surface will eliminate the possibility to develop an equilibrium between the molecule concentrations inside and outside of the implant material. As an example, nylon has been found to absorb water from the surrounding environment when implanted (Edwards, 1983). The water molecules act as plasticizers, which reduce the cohesion and bonding between the chains of the polymer. The final result is a reduction in strength.

Whether property degradation will be a determining factor in material selection for vascular implants depends largely on the planned time of implantation for the device. For a graft that will permanently replace a section of blood vessel, it is extremely important to maintain the tissue properties at an appropriate level. However, many blood contacting applications involve short-term use—for instance intravenous catheters for administering blood or pharmaceuticals. In these cases, the length of intended use is typically less than a few days, and the catheter can be relatively easily replaced if necessary. Therefore, for short-term implants, the prevention of blood clot formation and blood cell damage, along with ease of use and cost, become the determining factors in material selection.

3.3 Current Material Selection

Since the 1950s, polymer fabrics have been the primary material used for long-term vascular grafts. Nylon was introduced in 1955, but was withdrawn from use after the occurrence of aneurisms within the grafts indicated a loss of mechanical integrity *in vivo* (Edwards, 1983). Teflon (PTFE, Gore-Tex) and polyethylene terephthalate (PET, Dacron) have been shown to have acceptable amounts of property degradation when implanted for 20 years or more (Snyder, 1983). Table 5 shows results on the loss of strength in various polymers that were implanted for 100 days. The Dacron showed an initial drop in strength that then stablized at an acceptable level.

Typical vascular grafts are constructed of a woven or knitted fabric and are crimped, to both prevent kinking and to allow for longitudinal expansion (Fig. 8). The response of the graft *in vivo* will depend not only on the constituent material selected, but also on the weave of the fabric and on its processing (Sawye, et al., 1983). Grafts are typically tested to validate their tensile or bursting strength, and the values obtained for the constructed vessel will differ from those of the bulk material due to the knitted or woven nature of the fabric.

Table 5 Average Changes in Tensile Properties of Synthetic Grafts Implanted for 100 Days

Material	Loss of Strength (%)
Nylon	−81
Orlon	−6.9
Dacron	−10.1
Teflon	+3.2

Source: Data from (Edwards, 1983)

Pyrolitic or LTI (low-temperature isotropic) carbon has been found to have excellent anticoagulation properties (Bruck, et al., 1973), without the need for heparin coating of the material. The original LTI formulation required a solid substrate and so was more conducive for use on shunts or leaflets of heart valves. A newer, ultra-low-temperature iostropic carbon (ULTI) can be vapor deposited onto fabrics as well (Bruck, 1980), allowing for its use in regions that require flexibility (Fig. 8). ULTI carbon, which can be deposited with a thickness of less than 1 μm, has been shown not to affect the compliance of the underlying fabric (Sharp, 1983), indicating that it will maintain its flexibility when deposited on a fabric graft.

For short-term implants, generally in the form of catheters, silicone rubber has become the standard. Its optical translucence makes it easy to monitor fluid transfer, it is easy to use, and it is highly biocompatible.

Fabric grafts and silicone tubes, however, are not adequate for the small-vessel replacement that may be required during reconstructive surgery. As a result,

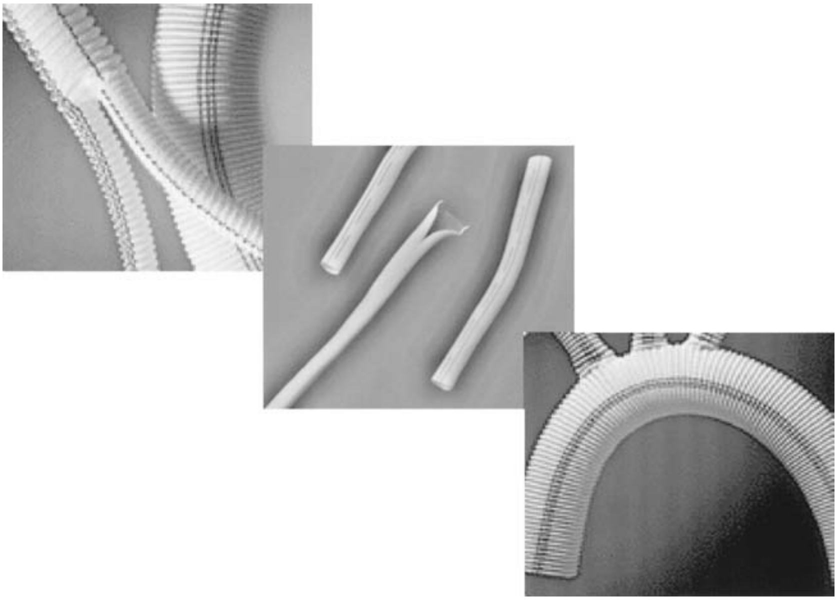

Fig. 8 Left and right: Examples of fabric arterial grafts, illustrating the crimped structure that prevents kinking *in vivo.* Center: Extruded PTFE graft lined with pyrolytic carbon.

tissue engineering techniques are currently being employed to construct small-diameter vessels from natural materials, such as chitosan (Chupa, et al., 2000). Research such as this is working to expand the possibilities for material selection, not only for vascular prostheses but also for all biomedical implants.

4 SPACE-FILLING BIOMATERIALS: BREAST IMPLANTS

Implants designed to fill voids where tissue has either been removed or destroyed are commonly used in reconstructive surgery. Hard tissue, soft tissue, and combination materials have been utilized to replace or augment cranial defects, the loss of an ear, nose, or eye due to trauma or disease, and congenital facial abnormalities, among others. Perhaps the most familiar and infamous space-filling implant was, and is, the breast implant. Originally designed for reconstructive surgery following mastectomies, breast implants became increasingly popular for cosmetic enhancement during the 1970s and 1980s. The question of whether the implants were the cause of the illnesses and disabilities reported in the 1990s has never been answered to everyone's satisfaction, and probably never will be. It is an example of a biocompatibility issue that, if true, went unnoticed for many years. It also serves notice to all biomedical implant designers that because an implant does not indicate any problems after 5 years does not mean that monitoring of potential complications should stop.

Breast implants were designed with one function in mind—to replace tissue that had either been removed through surgery or that an individual felt nature had left lacking. There was no other physiologic role for the implant, which made its design much simpler. The first breast augmentation utilized injections of silicone, paraffin wax, or bees wax directly into the tissue surrounding the breast. This method was banned by the Food and Drug Administration (FDA) in the 1960s as the injected material was seen to migrate and lose its shape. In addition, because of the large contacting surface area between the injected material and surrounding tissue, adverse tissue reactions were seen (Frisch, 1983). Since that time, breast implants have utilized confined volumes of materials (Figure 9).

4.1 Function

Space Filler

As stated above, the single role of a breast implant is to fill up a given volume of space within the body. However, cosmetic appearance and "feel" have also dictated much of the development of the implants. The density of the implant should be similar to or less than the surrounding tissue, so that tissue damage does not occur due to increased weight. The consistency of the material should also be somewhat similar to the composite of fatty and connective tissue that it has replaced. Thus, metals, ceramics, and solid pieces of polymer would be inappropriate.

4.2 Biocompatibility

Capsule Formation

No artificial material that is implanted into the body will be ignored completely. The degree of the reaction from the immune system will depend on a number

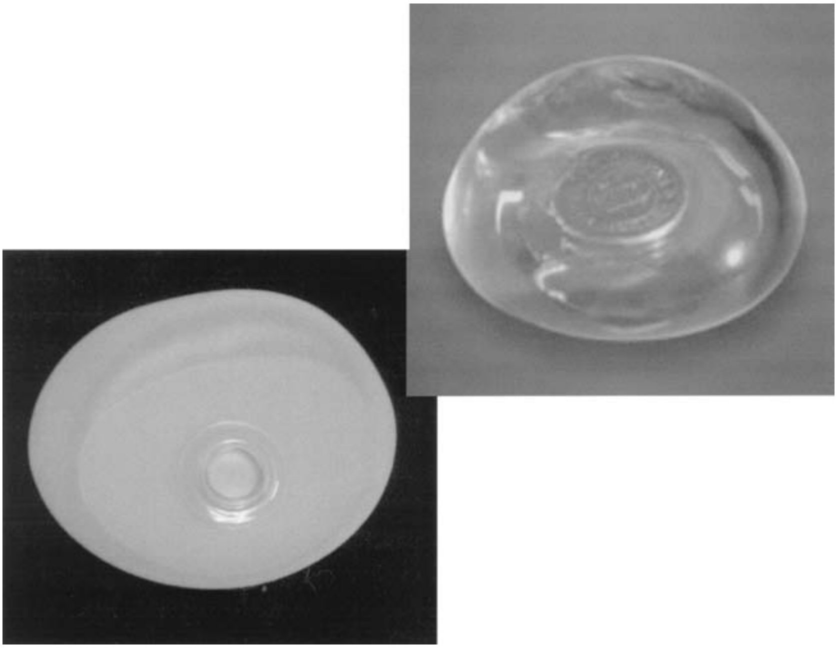

Fig. 9 Examples of saline-filled breast implants showing the silicone elastomer envelope. Surfaces can either be textured (upper) or smooth (lower). The textured design is thought to reduce the incidence of capsular contraction.

of factors, including the chemical or electrical activity of the material. The initial reaction of the body to a foreign object is to wall it off. A fibrous capsule—a result of the normal wound healing response of the body—will develop around the structure, with the thickness of the capsule increasing in regions of high chemical activity, high electrical activity, or sharp corners. The presence of this fibrous capsule formation can be taken advantage of to assist with implant fixation, however.

For objects with smooth surfaces, the fibrous capsule will simply grow around the outer surface of the material. If the surface of the foreign body is porous, however, fibrous tissue will begin to grow into the interconnected pores of the structure. For bony implants, this tissue can become calcified and develop into a bony junction between the implant and the surrounding tissue. For soft tissue implants, this fibrous capsule can become integrated with the surrounding connective tissue of the body to provide firm anchorage of the implant at a desired location. Complete fibrous tissue integration with the implant material will be most efficiently achieved with a pore size ranging from 5 to 30 μm (Rose and Litske, 1989; Wilkerson and Zalina, 1994).

The amount of desirable fibrous capsule integration with the implant must, however, be assessed and the area of porous surface determined as part of the overall design. One breast implant design that failed because of an overabundance of fibrous capsule integration was the sponge design developed in the 1950s (Dukes and Mitchley, 1962; Edgerton and McClary, 1958). This implant, which met the space-filling requirements and was composed of polyvinyl alcohol foam, was found to experience complete fibrous ingrowth. Although the implant was firmly fixed in the correct anatomical position, the fibrous capsule tended to contract and calcify, resulting in a condition known as marble breast syndrome.

All breast implant designs have shown some incidence of capsular contraction, which in severe instances has required the implant to be removed. This has led to the FDA requirement in the United States that all implant manufacturers inform prospective patients of this potential complication and the fact that breast implants should not be considered to be lifetime prostheses. While no implant design has been developed that eliminates this phenomenon, the area of fixation patches on the posterior surface has been reduced, textured (though nonporous) surfaces have been introduced for the silicone envelope, and clinical interventions, including massage, have been prescribed in attempts to minimize the problem.

Carcinogenesis and Immunological Complications

For both carcinogenesis and general immunological considerations, it is important to examine the response of not only the bulk material that is to be used but also the components of the materials. This includes unreacted monomer, plasticizers, fillers, and any products that can result from the metabolic breakdown of the bulk material *in vivo*. Any chemical compound that could be released by an implant is a potential hazard. The complexity of this analysis becomes readily evident.

The exact mechanism, or mechanisms, by which healthy cells in the body mutate into malignant cells and cause cancer is not fully understood. However, it is a well-accepted principal that all possible attempts should be made to avoid implanting a material into the body that would increase the risk of cancer. As cell mutations and tumor development can take many years to become evident, how can this be done practically for newly developed materials?

Carcinogenesis, or the production of cancer, can occur in the immediate vicinity of an implant, due to the presence of various chemicals, or at a remote location in the body, due to the transport of released materials. Materials in the body can act in one of three ways to cause cancer: (1) as a complete carcinogen, resulting in cancerous changes by itself; (2) as a procarcinogen, a benign chemical that is metabolically modified into a carcinogen by the body; or (3) as a cocarcinogen that is not likely to itself cause cancer, but will increase the activity of a complete or procarcinogen with which it comes into contact. It is hypothesized that there is no threshold below which a carcinogen present in the body is completely safe; however, as concentrations of a chemical increase, the probability that they will induce cellular changes that result in cancer also increase (Black, 1999). A tumor may also develop in response to the presence of a solid foreign body made of a noncarcinogenic material, due to the disruption of the normal environment of the surrounding tissues (Black, 1999).

Testing of materials for their carcinogenic potential ranges from cell culture studies that examine whether a material causes cells to mutate (Forster, 1986), to *in vivo* animal tests and the development of databases to track the occurrence of tumors in implant recipients. There are drawbacks to all of these tests, and none, with the exception of a longitudinal study of tens of thousands of patients with the same implant, can definitively determine if there is a link to cancer for an implant or a material that does not initially appear to be carcinogenic. The primary benefit of these tests is to eliminate from consideration materials that appear to have a high likelihood of causing tumors. General guidelines for basic biocompatibility testing have been issued by the International Standards Organization and other regulatory agencies (ISO-10993, 1992–2000).

Similar problems exist for the documentation of other systemic or remote tissue complications. Techniques have been developed to evaluate the immunological response to a material (Marritt, 1986); however, the link between local immunological sensitivity and a systemic autoimmune reaction may not be easily discernable. It is important to determine whether the occurrence of a particular disorder, whether it is lymphoma or rheumatoid arthritis, is occurring within the implanted population at rate that is higher than the general population. One of the difficulties in tracking the incidence of these occurrences is the fact that many implant materials and combinations change on a time scale that is short compared to the development of cancer and other immunological conditions. As these material combinations change, it introduces another variable that requires a higher number of subjects before a statistical determination can be made. However, the establishment of databases at the national, or preferably international, level for tracking of all complications associated with biomedical implants will allow for a more complete analysis and more rapid understanding of the problems associated with this imperfect science.

4.3 Current Material Selection

Prior to 1992, the most common form of breast implant involved a small bag of silicone elastomer filled with silicone gel. Originally introduced in 1962 (Cronin, 1983), this implant was popular among patients and physicians alike. While the silicone elastomer of the bag has been utilized in biomedical implants for many years without significant adverse effects, the breast implant scare of the 1990s was based on rumors that the silicone gel had a tendency to leak from the surrounding bag and cause adverse reactions at both local and remote locations. Since that time, the majority of breast implants have utilized saline within the same silicone rubber bag. If the saline does leak, or the implant does rupture, the reaction with the tissue will be completely benign. This design has been an option since 1965 (Rubin, 1983); however, most patients opted for the silicone version due to its improved cosmetic appearance.

5 SUMMARY

There are few, if any, current implants that can be described as perfectly meeting their design goals and constraints so that no further investigation of design or material selection is warranted. As materials continue to be developed, whether specifically for biomedical applications or in some different discipline, the selection of biomaterials for implants will remain a challenge in the design of the optimum implant. The evolution of tissue engineering from a bench-top science to a clinically workable tool for new implant design will also open new doors for the development and use of biomaterials. In all of these cases, however, the same principles apply to the selection of a material for a biomedical application. The selection process can be summarized in the following way:

1. Determine the functional requirements of the material for the particular application (preferably with an idea of the overall design in hand).
2. Select a group of materials that appear to meet those functional requirements and ensure that all confirming tests are conducted in an environment that simulates human physiology.
3. Determine the biocompatibility of the materials in terms of material degradation, tissue effects, blood compatibility, implant fixation, and long-term physiologic consequences.
4. Complete the design and approval process, with mechanisms in place to obtain data on functional or material complications for many years after clinical use is initiated.

As with all aspects of engineering design, selection of materials for biomedical implants is like solving a puzzle—given all of the goals and constraints, what is the one solution that best answers the question that has been posed? And if a current solution does not exist, can one be developed? Some may argue that careful selection of materials for medical applications is more important than for other realms of engineering, due primarily to the overriding fear most people have of disease or injury. Others may disagree. It is certain, however, that development, evaluation, and selection of biomaterials is one of the most challenging areas of materials science. No two physiologic environments are exactly the same; therefore, no two implants will respond in exactly the same way. It is also much more difficult to predict the loading, chemical, and biological environment that an implant will see in a particular individual than it is to model the potential variations in loading of a car or a building. The science of biomedical materials has improved tremendously since the days of George Washington's wooden teeth—and the development of new materials has just begun.

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CHAPTER 38

SELECTING MATERIALS FOR MEDICAL PRODUCTS

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1 INTRODUCTION

Medical device and medication delivery system products are critical sectors in the medical, pharmaceutical, and health-care industries. Many medical devices and solution drug delivery systems are disposed after a single use. These disposable devices and delivery systems are generally produced from polymeric materials. The disposable materials that host solution drugs or biological agents are recognized as parts of drugs and biological agents. New drug applications (NDA) to the Food and Drug Administration (FDA) and to foreign government regulatory agents are required. In contrast, the materials used in many devices

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Handbook of Materials Selection. Edited by Myer Kutz

and disposables that have short solution drug or human contacts require 510K approval.

A new medical product must prove product safety and efficacy to the FDA before market launch. Material toxicology such as testing under U.S. Pharmacopoeia (USP), European Pharmacopoeia (EP), or Japanese Pharmacopoeia XIV (JPXIV) guidelines is used to ensure the safety of the materials for medical product applications. The extractive contents and levels from poststerilized materials indicate the potential impact to solution drug or biological agent quality. Solution stability is used to understand the interactions of polymeric materials with the hosted medication fluids during product shelf life. Examples in Table 1 of medical devices include angioplasty, hemodialyzer, syringe, and oxygenator. Disposable sets and solution containers in medication delivery system include those used to host intravenous (IV) solution, blood components, peritoneal dialysis solutions, parenteral nutritious solutions, and biological agents for infusion and transfusion therapies.

Selecting materials for medical device and disposable products have stringent guidelines. Table 2 lists the FDA International modified ISO 10993-1 test matrix. The materials are tested after exposure to the desired sterilization modes. The biological testing on the materials is dependent on the intended contact duration and body contact. The contact duration is classified as limited (<24 h), prolonged (24 h to 30 days), and permanent (>30 days). The body contact is categorized according to surface devices, external communicating devices, and implant devices.

Regardless of the applications of medical products, the health-care and medical industries always attach a premium to medical product optical clarity. A material's ability to allow unimpeded light transmission is not just a desire to be "clean" but is rather rooted in good clinical practice. For example, in nu-

Table 1 Examples of Medical Products

Medical Product	Product Characteristics
Angioplasty, hemo dialyzer, and oxygenators	Medical device whose materials are compatible to blood and blood vessel tissue.
Intravenous (IV) solution and drug delivery system	Solution drugs stored in a medical container whose materials are suitable to host solution drugs for long shelf life storage without concerns of drug interaction and concentration variation.
Blood collection, processing into red blood cells (RBC), platelets, and plasma, and then storage for blood transfusion	Anticoagulants are stored within blood collection bags to prevent blood agglomeration. A breath container material is a must for red blood cells and platelet storage. Low-temperature ductile impact film for plasma storage at subambient temperatures.
Nutritious solution and peritoneal dialysis (PD) solution containers and their delivery system	Solution drugs stored in medical containers that can be steam sterilized to meet sterility requirements. No drug degradation or harmful extractives from container materials during autoclaving.
Pharmaceutical blister, bottles, and containers	To host solid drug tablets whose packaging materials have good barrier properties of moisture, O_2, and CO_2, and UV exposure.
Syringe and drug prefilled syringe	Empty is a medical device that requires on 510K approval, but prefilled syringe is a drug product that require New Drug Application (NDA) approval.

Table 2 FDA-Modified ISO 10093-1 Test Matrix

Device Body Contact	Contact Duration[a]	Cytotoxicity	Sensitization	Irritation / Intracutaneous	Systemic Toxicity	Subchronic Toxicity	Genotoxicity	Implantation	Hemocompatibility	Chronic Toxicity	Carcinogenicity	Repro. / Devel. / Biodeg.
Surface devices												
Skin	Limited	X	X	X								
	Prolonged	X	X	X								
	Permanent	X	X	X								
Mucosal membrane	Limited	X	X	X								
	Prolonged	X	X	X	○	○	—	○				
	Permanent	X	X	X	○	X	X	○	—	○		
Breached or compromised surfaces	Limited	X	X	X	○							
	Prolonged	X	X	X	○	○	—	○				
	Permanent	X	X	X	○	X	X	○	—	○		
External communicating devices												
Blood path, indirect	Limited	X	X	X	X	—	—	—	X			
	Prolonged	X	X	X	X	○	—	—	X			
	Permanent	X	X	○	X	X	X	○	X	X	X	
Tissue / bone / dentin communicating	Limited		X	X	○							
	Prolonged	X	X	○	○	○	X	X				
	Permanent	X	X	○	○	○	X	X	—	○	X	
		X										
Circulating blood	Limited	X	X	X	X	—	○	—	X			
	Prolonged	X	X	X	X	○	X	○	X			
	Permanent	X	X	X	X	X	X	○	X	X		
Implant devices											X	
Tissue / bone	Limited	X	X	X	○							
	Prolonged	X	X	○	○	○	X	X				
	Permanent	X	X	○	○	○	X	X	—	X	X	
Blood	Limited	X	X	X	X	—	—	X	X			
	Prolonged	X	X	X	X	○	X	X	X			
	Permanent	X	X	X	X	X	X	X	X	X	X	

Note: X = required; ○ = option.

[a] Limited contact, ≤24 h; prolonged contact, 24 h–30 days); permanent contact, >30 days).

merous primary medical containers, prior to administration of the medical fluid to the patient, the nurse is required to inspect visually for particulate matter (PM) contamination. This inspection is required to protect patients against the danger of exposure to the visible contaminants.

In situations where medications in the powder form need to be mixed or compounded prior to administration, the ability to visually inspect for the completion of dissolution is another important safeguard for patient safety. Recently, transfusion therapies involving harvested, stored, and cultured cellular components became widespread. During the *in vitro* phase, these cellular components frequently need to be manipulated through separation, fractionation, and incubation for diagnostic or therapeutic purposes. In many of the steps, the ability to inspect contents visually or microscopically is crucial to the clinical protocol. These are just a few reasons that optical clarity is a prerequisite criterion for medical material selections.

Another attribute specific to the health-care and medical industries is the need to deliver therapies in a sterile manner. As a consequence, compatibility with common sterilization methods is a must. These methods include steam autoclaving, ethylene oxide (ETO) sterilization, and ionizing radiation (either gamma or electron beam.)[1,2] Steam autoclaving places a severe burden on the prospective medical material with the use of high temperature (such as 121°C) and pressure. Ethylene oxide is currently being phased out due to worker exposure and environmental concerns, while long-term shelf life stability is an issue for the material postradiation sterilization.

A third requirement that has received increasing attention is environmental compatibility. As professionals concerned with health care, we obviously cannot, in the course of mending our patients, destroy the environment that we live in and our children will inherit. Of course, the quantitative ‘‘environmental score’’ is quite elusive and often subjected to passionate debate. However, some of the basic tenets are very obvious; progressive companies have already adopted many of the practices in this direction.

All these have urged the medical industry to select safe, clean, and environmentally friendly materials to meet safety, manufacturability, and functionality requirements for medical products. These requirements thus pose the constraints and challenges on the materials selected during product development for medical, pharmaceutical, and health-care industry.

2 CHALLENGES OF MEDICAL PRODUCTS

Medical product challenges faced today are not only from technical advances but also from business competition. Safety is the highest priority. Product clarity, stability, biocompatibility, and low extractives are highly valued. Many medical and health-care products require a specific level of oxygen (O_2), carbon dioxide (CO_2), or water vapor transmtission rate (WVTR) at room temperature. These products can be either very rigid or very flexible, and for some drug delivery systems and medical devices superior subambient impact is required. Furthermore, the selected medical materials should be compatible to the desired sterilization mode and render no harmful damage to the packed devices, drugs, and disposable systems.

Polymeric materials are part of the finished medical products. A lowest possible total system cost is used to direct product development and finished product manufacturing. From a business viewpoint, the lowest system cost guideline has a great impact in determining product design, material selection, and manufacturing processes. The lowest system cost can be achieved when the design, materials, and manufacturing are taken into accounts simultaneously. For example, a lowest system cost is still feasible when employing a costly material that enables to simplify the design and reduce manufacturing cost.

In the managed-care environments, the patient care has been gradually switched from doctor care to insurance provider care. The cost is extensively monitored and supervised. Under this great competition, high medical product quality is expected with no further premium increments. Moreover, home care also gradually becomes popular. This requires the medical manufacturers to design ease-of-use medical products for patients with no specific training in the medical field. The medical design and materials selection can play a critical role to fulfill the patient expectation, meet product functions, and achieve business goals.

3 PRODUCT DEVELOPMENT FUNDAMENTAL FACTORS

The key factors that govern the development of medical products can be categorized into four distinct areas: product design, material selection, manufacturing process, and product performance.[3,4] The detailed requirements of these four areas are listed in Table 3. Designing user-friendly products, selecting high-performance low-cost materials, establishing cost-effective manufacturing processes, and validating product quality for safety and efficacy are a must. These considerations can greatly restrict a manufacturer's choices for developing a product.

3.1 Product Design

Medical product design is focused on safety and efficacy. Product design begins with concept design, design drawing, and stress analysis and ends with the evaluation of the prototype. Different design iterations are created and materials are selected to build prototypes. The prototype is modified after feedback from clinicians, patients, engineers, and manufacturers. The functionality is tested to confirm the desired efficacy.

For example, to maintain product sterility integrity during shelf life, a closed system design is required. In addition to product design for safety and efficacy, the product design also includes device component design such as molding and assembly, particularly for component joining, welding, and bonding. These designs are typically under the constraints of materials availability, cost of materials, materials compatibility for joints, manufacturability, sterilization modes, and product integrity during the shelf life and up to the time of use. Moreover, marketing always prefers to have features beyond safety and efficacy; good product features are usually an excellent vehicle to win patient acceptance.

3.2 Selecting Materials

The process of selecting suitable materials for medical products begins with the creation of a precise and accurate definition of the product's material and functional requirements. Finding the right polymers for medical products requires simultaneous consideration of design, processing, and performance needs.[1] Other critical factors considered at the material selection stage include biocompatibil-

Table 3 Medical Product Development Considerations

Product design	Safety and efficacy Product functionality Ease of use Product integrity Design a closed system to ensure sterility integrity Flexibility for medical product design Easy assembly No build-in residual stress in plastic components Bonding/assembly capability among product components Easy quality control by operator vision or instrumental sensor Sterilize/form/fill/seal
Materials selection	Meet requirements of safety, design, processing, and performance Material compatibility for product components assembly Drug and solution contact Biocompatibility and chemically inert Leachables and oligomer residues Optical clarity WVTR, O_2, and $C0_2$ barrier Subambient impact resistance Material aging, particularly after sterilization Additive chemicals and catalyst residue Lot-to-lot consistency from resin supplier Environment friendliness Cost Supplier technical service
Manufacturing processing	Extrusion/molding/thermoforming capability Large-scale manufacturability High production output rate Wide processing operation window Compatibility with the plant's existing manufacturing systems Assembly technology Sterilization methods
Product performance	Safety, efficacy, and quality Unique features Cost/performance Function oriented Market competition Customer delight User friendly Cosmetic appearance Touching feeling Odor

ity, leachability, drug–plastic interaction, oxygen and moisture barrier protection, optical clarity, ultraviolet (UV) stability, shelf life, the end-use environment, and total system costs. In addition, designers must consider the demands of downstream operations such as component bonding, assembly, shipping, storage, and post-use disposal.

One of the key factors in medical material selections is the sterilization effect to the material properties. Steam sterilization demands that the melting points of materials exceed their autoclaving temperatures. Ethylene oxide (ETO) sterilization needs to vent the residual ethylene oxide from the device fluid path to a minimum level before products can be released. Radiation poses potential materials degradation that could impact the product performance and also its extractives might interact with solution drugs or biological agents.

Gamma Radiation Effect on Medical Materials

Gamma sterilization is becoming popular in the medical device and packaging industry because of convenience and low cost. Concerns of worker exposure to ethylene oxide and temperature limits of medical materials during high steam autoclaving have made gamma sterilization more preferable. This mode of sterilization is a consequence of the high-energy electrons released from the interaction of the gamma-ray photons with materials. These high-energy electrons in turn react with the deoxyribonucleic acid (DNA) sequences in the microbiological burden in medical devices and drug delivery systems and permanently alter their chemical structures to render them innocuous.[1]

The high-energy electrons, however, can also initiate ionization events in the material being sterilized. It can create peroxy and hydroperoxy free radicals in the presence of oxygen and start the degradation cascade. Different materials degrade via various mechanisms, leading to different modes of failure such as discoloration, excessive pH shifts and high extractables, and catastrophic failures. Gamma exposure at 20 and 50 kGy (10 kGy = 1 Mrad) is usually used for radiation sterilization. Oxidative induction time (OIT) and yellowness index (YI) are used to identify a material's radiation sterilization compatibility. OIT, a method of thermal analysis measured by differential scanning calorimetry (DSC),[2,5–8] is used to measure the total stability of a polymer at a given condition. A more stable plastic has a higher OIT value.

Polyvinyl chloride (PVC) thermal stability, measured as OIT in Fig. 1, has been reported to exhibit a rather sharp maximum with respect to the concentration of a primary stabilizer, calcium zinc stearate at about 0.2 phr (part per hundred) or about 0.13% for the system studied.[5] It was noted that an extremely linear relationship was found for the secondary stabilizer such as epoxidized oil. Combined together, Fig. 2 shows the PVC stability function spanning a three-dimensional design space.

The OIT responses of a PVC formulation at 230°C as a function of the sterilization dose is shown in Fig. 3 with a dose rate of about 6 kGy/h. From the

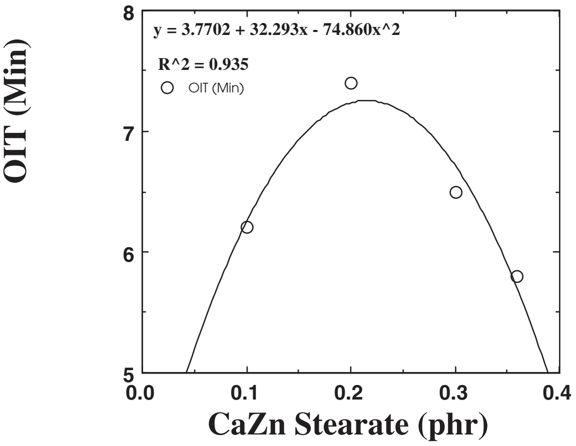


Fig. 1 PVC OIT dependence on CaZn stearate.

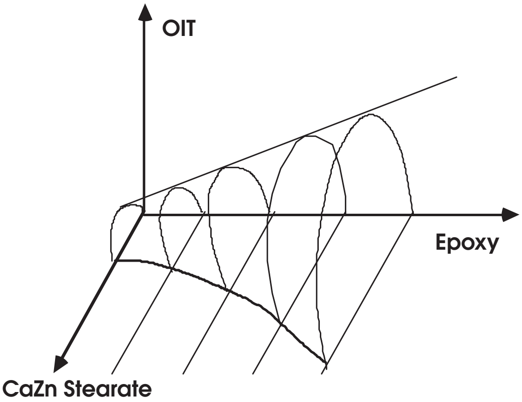


Fig. 2 Three-dimensional schematic of PVC stability.

zero dose OIT of about 9 min, a steady reduction in total stability was seen. In addition, the slope (rate of OIT decrease) appeared to increase from 20 to 40 kGy, indicating a nonlinear response accelerating the degradation reaction at high doses. Figure 3 also indicates that after 40 kGys the formulation loses more than half of the initial total stability. This will limit the maximum dose this particular formulation was capable of sustaining.

Radiation also changes polyolefin stability. In a hindered phenol-stabilized high-density polyethylene (HDPE) film system, the film OIT at 200°C was determined for dose levels of 0, 20, and 40 kGy. Results in Fig. 4 clearly indicated significant annihilation of the antioxidants during the course of the sterilization. It was noted that the OIT was nearly flat at 20–40 kGy. This suggests that the antioxidant in question did not provide additional protection when the radiation dose was reduced from 40 to 20 kGy.

When Figs. 3 and 4 were compared, PVC was noted to have a slower stability reduction than HDPE when subjected to radiation. In addition to the inherently

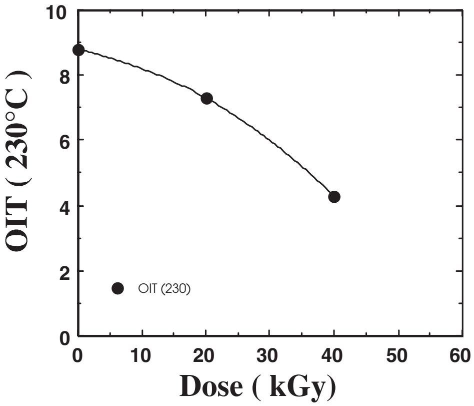


Fig. 3 Effect of radiation dose on PVC stability.

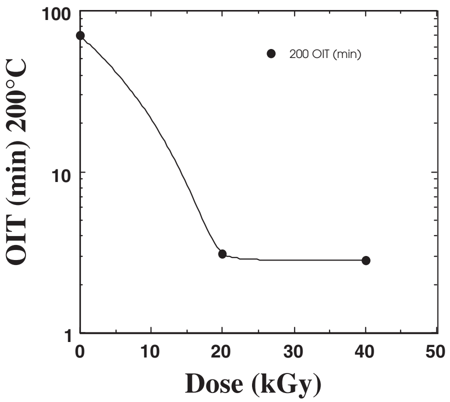


Fig. 4 Effect of radiation dose on HDPE Stability.

more stable of PVC over PE, it can be also due to the higher concentration of secondary stabilizer in PVC, which can be about 10%, as shown in Fig. 5. This may explain why the medical products made from PVC seldom encounter catastrophic failure after radiation.

Figure 5 also shows that radiation sterilization reduced the PVC secondary stabilizer concentration, as measured by high-pressure liquid chromatography (HPLC). This in turn reduced material stability as evidenced by OIT. Sterilization by radiation was noted to deplete more secondary stabilizer than the subsequent steam autoclaving and pasteurization combined.

In contrast, catastrophic failures have been reported during polypropylene (PP) shelf life storage. Intense investigation came to the following consensus: [5,9–12] The long-lived free radicals trapped in the crystalline domains migrating toward the crystalline/amorphous interface combining with available oxygen form peroxy and hydroperoxy radicals that initiated degradation near the interface.[3] As enough tie molecules between crystallites were cut through the chain

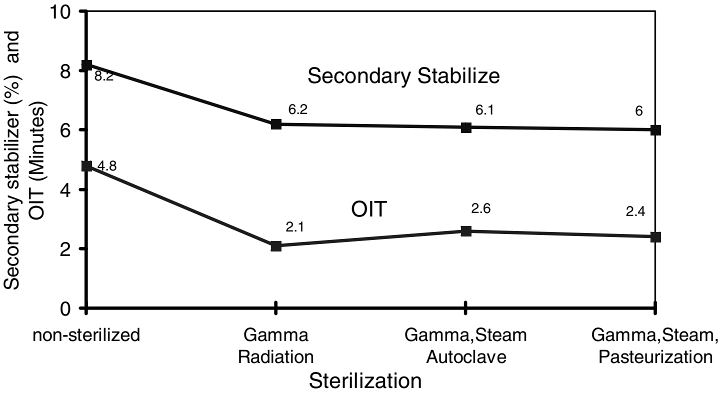


Fig. 5 Change of OIT with secondary stabilizer concentration of PVC.

scission process, significant reduction of PP elongation could occur, which would lead to catastrophic failures.

Since the stabilizer molecules in PP reside primarily in the amorphous phase, its effectiveness to react with primary free radicals preferentially is an indication of the overall postirradiation stability of the material. Accordingly, it is interesting to assess the correlation between OIT and radiation dose based on different suppliers.

Figure 6 shows PP from three different suppliers and their OIT at 200°C at 0, 25, and 50 kGy of doses. It is clearly seen that supplier A's formulation, where the OIT vanishes after only 25 kGy, was not as stable or effective toward gamma radiation as the other two formulations. On the other hand, although PP formulations from suppliers B and C had experienced major reduction of OIT, significant portions remained to protect against further degradations.

Figure 7 plots the OIT from three PP suppliers with the phenolic primary antioxidant assayed by HPLC. The very good correlation indicated the promise of using this simple procedure as an early screening tool for evaluating PP radiation stability. The stability of a radiation-sterilized PP can thus be simply and very rapidly determined by OIT, and the remaining antioxidant level of sterilized PP can be easily obtained by the correlation similar to that in Fig. 7. Moreover, Fig. 7 indicates that the stability of sterilized PP depends on the supplier stabilizer system.

In an HDPE system stabilized with hindered phenolic antioxidants, the result of high-temperature induction time dependence on antioxidant concentration was established, as shown in Fig. 8. The exceptionally linear response of OIT at different temperatures for this system clearly indicates the potential as a simple (minimum sample preparation), and very rapid (minutes) nonspecific assay for evaluating HDPE radiation stability.

Yellowness index (YI) can be also used to characterize the material stability. A higher YI suggests more extensive degradation as the same family of materials is compared. When the YI of PVC film was plotted with OIT measured at 230°C, Fig. 9 shows a very good correlation. Both OIT and YI samples were taken from a 190°C oven heating test at different times. It is noted that as OIT reduced

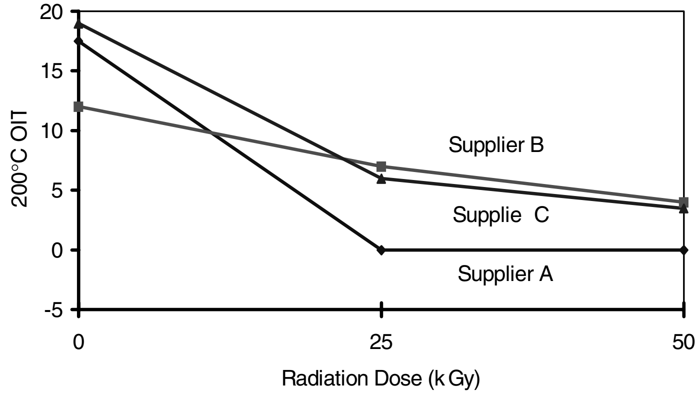


Fig. 6 Effect of radiation dose on PP from various supplies.

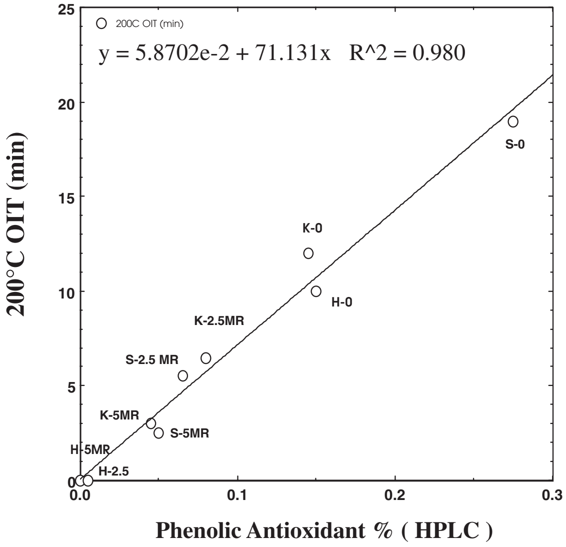


Fig. 7 PP OIT dependence on antioxidant concentration.

from 9 to 4 min., the PVC YI value increased from 20 to 50. The increase in YI can be related to the poly-ene formation in PVC and quinone and hydroquinone formation from phenolic antioxidants due to radiation and thermal exposures.

Without quinone and hydroquinone formation in a proper stabilizer system, the increase in PVC color, measured by YI, is the result of a series of conjugated dienes (poly-enes) in PVC molecule chains formed by cationic dehydrohalogenation of PVC. Since the electrons on these conjugated dienes can freely move over the entire length of the conjugated diene, a one-dimensional electron well resulted. The length of conjugated dienes can be increased due to ineffective

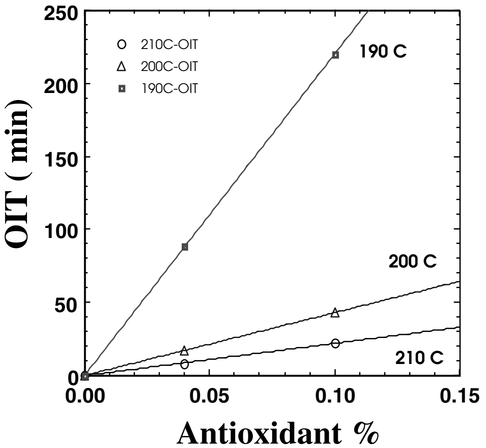


Fig. 8 HDPE OIT dependence on antioxidant concentration.

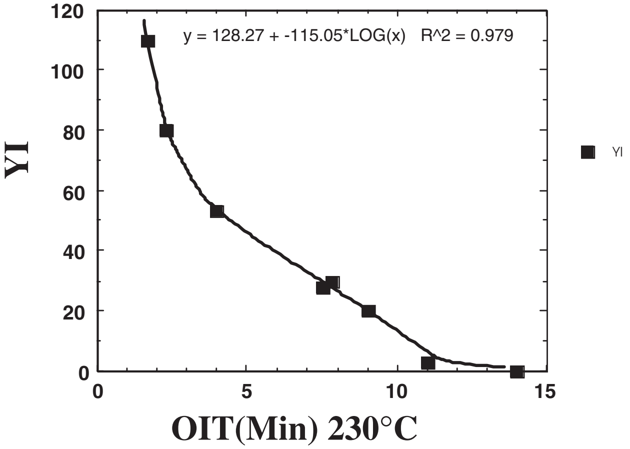
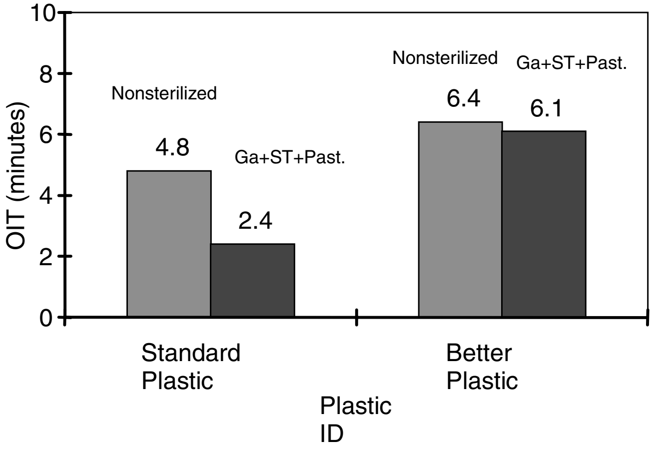


Fig. 9 Correlation of PVC OIT and color YI.

stabilizer protection during radiation sterilization. The energy levels and the absorption spectra of the free electron can thus begin to move from ultraviolet (UV) toward the visible wavelengths. As the absorption spectra increase in intensity from the short wavelength direction, the originally clear PVC film starts to appear yellow to dark yellow, orange, red, and finally black as the degradation progresses.

In PVC medical containers used for drug delivery system, a better quality film material made from a superior stabilizer system is expected to have a higher thermal stability than a standard PVC. The superior stabilizer system has an extra gamma-compatible additive component than the standard stabilizer system. Figure 10 shows that the better-quality PVC has a higher OIT for both sterilized



Ga+ST+Past. = Gamma + Steam autoclaving + Pasteurization

Fig. 10 Effect of stabilizer system on PVC stability.

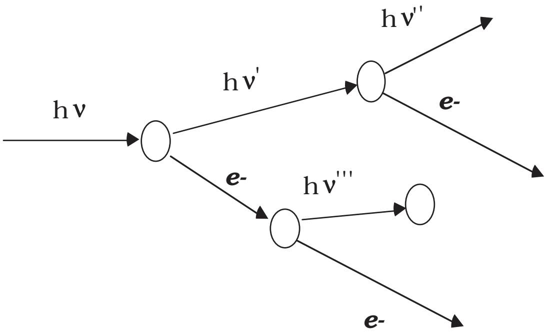


Fig. 11 Compton scattering with matter.

and nonsterilized material. This is because a more effective PVC stabilizer system can minimize both thermal degradation during film extrusion and the subsequent radiation sterilization.

Electron Beam (E-Beam) Radiation Effect on Medical Materials

Recent advances in electron beam technology have made this mode of sterilization a worthy competitor to the traditional gamma processing. Some key factors of E-beam critical to medical industry are the increased available energy, compact design, improved reliability, easy turn on and off, and an effective source without steadily depleting with time.

The primary interaction between gamma radiation and E-beam with matter is known to be different. However, the major interaction is still Compton scattering for both cases. It is mainly the shower of secondary electrons, as seen in Fig. 11, that initiates the ionization events that activate numerous chemical reactions, many of which lead to oxidative degradation.

Although the main interaction with matter is basically the same for gamma and E-beam, minor differences between the two modes remain. The principal differences in Table 4 are the charge and the rest mass.[2] The absence of both a rest mass and a charge gives gamma radiation far greater penetration power than E-beam, whose penetration is primarily dependent on kinetic energy or the potential difference through which the electrons were accelerated.

Doses higher than those employed for sterilization were used to explore and accentuate minor differences. However, doses higher than that of the sterilizing

Table 4 Characteristic Comparison of Electron Beam and Gamma Irradiation

Mode	Electron Beam	Gamma Irradiation (^{60}Co)
Charge	-1	0
Rest mass	$9e^{-28}$ g	0
Energy	0.1–15 MeV	1.17 MeV
Velocity (C, light speed)	0.3–0.99 C	1.0 C

Note: Bond energy = 3–10 eV
UV source = 4–5 eV

dose are commonly encountered in medical device manufacturing. Rework can require a doubling of the radiation doses. Dose variations under a given exposure condition would also increase the upper dose limit to achieve the minimum dose required. Also, biological indicators with more resistant strains would likewise increase the required nominal dose.

A gamma irradiator typically delivers dose at rates approximately between 5 and 20 kGy/h, while for electron accelerators it could deliver dose rates as much as 10,000 times higher. Under such high dose rate conditions, significant thermal effects could arise to modify the material's reaction pathways. Due to the huge dose rate disparity, irradiation exposure times are also vastly different. While it is not uncommon for a gamma facility to deliver the sterilizing dose in several hours, the E-beam would take mere seconds for the same dose delivery. It is known that available oxygen diffusion during the exposure times would constitute another factor on materials degradation.[13]

The OIT conducted at 230°C for the gamma-irradiated flexible PVC in Fig. 12 exhibits a near monotonic reduction from the control. This PVC has a better thermal sterilizer additive packaging than the PVC reported in Fig. 3. Between 60 and 100 kGy, a slight reduction in slope is also apparent. However, when compared with the E-beam samples in Fig. 12, the gamma samples appear to follow a slightly steeper slope, at least for the lower dose exposures.

At or around 60 kGy, both the gamma and E-beam samples exhibited an upward shift in slope, indicating that additional doses have proportionally less degradation effect. This could be due to a secondary stabilizer, epoxidized oils, in the flexible PVC that reduces degradation through secondary reaction pathways. It is also interesting to note that even at 100-kGy dose levels, a significant fraction of stabilizer in the flexible PVC remained. This could be due to the relatively high stabilizer loadings for this medical PVC formulation.

In sharp contrast with the flexible PVC, the medium-density polyethylene (MDPE) film in Fig. 13 underwent a drastic reduction in OIT or antioxidant potency. At a dose of 30 kGy, OIT of MDPE was reduced from almost 900 min of induction time to about 20 minutes at 180°C. Subsequent additional doses resulted in a near linear decline in OIT on the logarithmic scale.

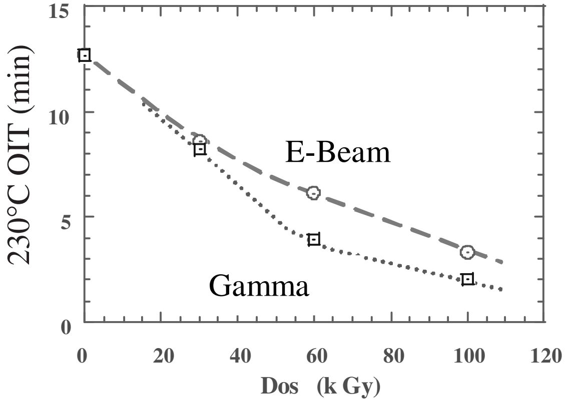


Fig. 12 Comparison of gamma and E-beam irradiation on OIT of PVC.

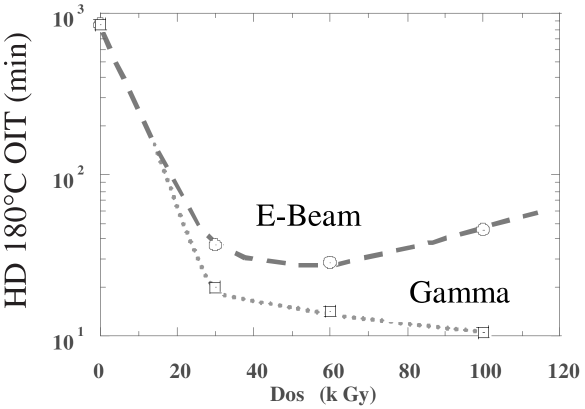


Fig. 13 Comparison of gamma and E-beam irradiation on OIT of MDPE.

This significant reduction in OIT stability was also seen in Fig. 13 in the E-beam samples at 30 kGy, although a slightly better retention of antioxidants is seen with the E-beam. At 30 kGy, the E-beam has about 40 min on OIT as compared with 20 min for gamma. From 60 to 100 kGy in E-beam, a distinct upturn in OIT is plainly evident. This upturn is most likely caused by thermal and exposure time effects under high dose rates from limited oxygen availability and accelerated free radical termination reactions favored at higher temperatures.

For the PP sample, a drastic OIT reduction is evident again, with even greater depletion ratios under gamma irradiation in Fig. 14. Once more, the OIT upturn at higher dose rates are very pronounced under E-beam. Most likely, similar dose-rate-related exposure time and thermal effects are the causes.

The significant departure toward greater overall stability and the upturn at 100 kGy could be rationalized by the extremely high dose rate and the resulting temperature rise. At these high doses, E-beam dose rates of over 200 kGy per second or 720,000 kGy per hour are encountered, or about four orders of magnitude higher than that of the gamma irradiation. In terms of the irradiation time,

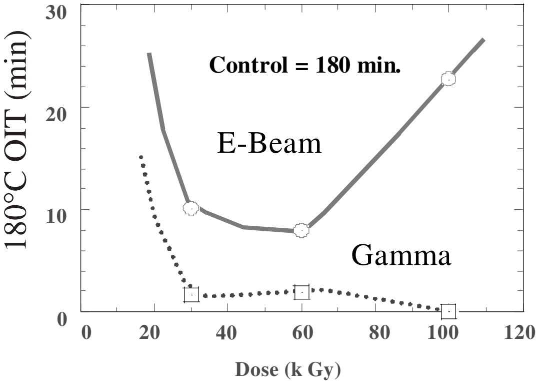


Fig. 14 Comparison of gamma and E-beam on OIT of PP.

instead of nearly 17 h for 100 kGy, it would take 0.5 s for the E-beam. These high dose rates would invariably lead to much higher free radical concentrations during the exposure and much higher recombination reactions discussed earlier.

Using a standard specific heat at room temperature of 1.7 J/°C for PP, and assuming no heat loss (adiabatic), temperature rises for PP are shown in Fig. 15. It is seen that partricularly for 100 kGy, a temperature over 85°C is reached. At the higher temperatures, the free radical recombination and termination reaction is expected to be much faster than at near 25°C. Less antioxidant consumption and less polymer degradation are predictable. The potential side effect of hihger dose rate by E-beam could be surface cosmetic defects on medical devices due to fast melt and cooling.

The E-beam results suggest medical device and medication delivery system made from PP materials could be E-beam sterilized. The concerns of castrophic failure could be minimized. The OIT indicates that PP has much better thermal stability at the high dosage of E-beam sterilization. This recommendation, however, still requires the confirmation from real-time sample testing, particularly with medical products for up to 2 years of product shelf life.

3.3 Newly Commercially Available Polyolefin-Based Materials

Recent progress in metallocene technology, including the ability to produce inexpensive metallocene catalysts, has led to the development of cost-effective metallocene-based polyolefin and cyclo-olefin materials. Metallocene polyolefins have the potential to achieve much better performance than existing PE and PP formulations. Cyclo-olefin materials give a high clarity for medical applications. Because they have properties similar to many specialty polymers and engineering plastics, metallocene polyolefins have the potential to replace PVC and some expensive engineering plastics, particularly for medical products requiring high clarity and impact strength and ductility at low temperatures. As a result, this polyolefin family is showing great potential for use in the medical and healthcare products industries.

The materials used by most of medical device and solution drug delivery systems today are still dominated by PVC materials. To be useful in medical

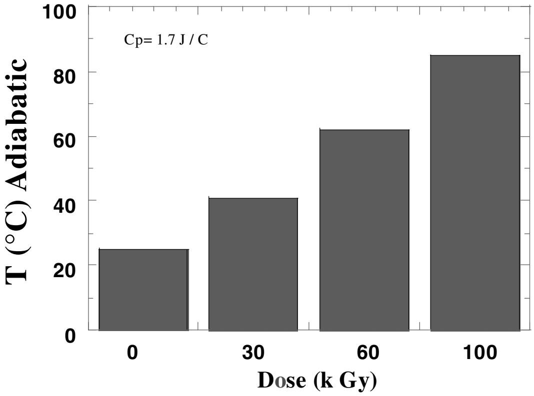


Fig. 15 PP temperatures rise at different E-beam doses.

products, however, metallocene-based polyolefins must also fulfill product design, processing, and performance requirements simultaneously. To meet these criteria with high product quality at the lowest possible cost, it will require a broad spectrum of material characteristics and processing capabilities.

PVC versus Metallocene-Based Polyolefins

Device manufacturers that wish to consider the use of metallocene polyolefins for use in medical devices or medical packaging will need to look at a wide range of characteristics. While the major traits of PVC have been established through a long history of use, those related to metallocenes are still emerging as the technologies develop and improve. Following are some of the key advantages and disadvantages of each, as their respective technologies now stand.

Advantages of PVC. Polyvinyl chloride can be used to produce a variety of medical products ranging from rigid components to flexible sheeting. The type and amount of plasticizer used determine the compound's glass transition temperature (T_g), which in turn defines its flexibility and low-temperature characteristics and thereby establishes its range of suitable applications.

Because rigid and flexible PVC components have the same material structure, they can be easily assembled by solvent bond. The two solvents most commonly used in PVC bonding are cyclohexanone and methyl ethyl ketone (MEK). Rigid parts that have been molded of PVC are suitable for ultrasonic bonding, while flexible extruded or calendered PVC films can be sealed using heat or radiofrequency (RF) sealing.

Medical products made from PVC can be sterilized by steam, ethylene oxide, or gamma radiation. Plasticized PVC can have a T_g as low as $-40°C$ and still be suitable for steam sterilization at 121°C. Additional characteristics that make PVC attractive include its low cost, broad T_g spectrum, wide processing temperature range, high seal strength, thermoplastic elastomer-like material properties, high transparency, wide range of gas permeability, and biocompatibility. Medical products made from PVC have passed critical toxicological, biological, and physiological testing. In sum, PVC is one of the best medical materials in terms of cost and function. No other single material has such broad material latitude.

Disadvantages of PVC. Even though many medical products have been made from PVC, the material continues to receive criticism.[14] The most commonly cited shortcomings involve toxic effluents produced during manufacture and the generation of hydrogen chloride (HCl) during incineration. Because HCl is a component of acid rain, postuse disposal costs for incinerating PVC can be quite high. Other concerns related to PVC depend largely on the type and amount of plasticizers used. For some PVC compounds, there is evidence of plasticizer leaching to medical solutions, chemical interaction with drugs, water vapor loss during long-term storage of medical solutions, and gas permeability.

Although these disadvantages sound challenging, most can be eliminated or managed using existing technologies. For instance, current PVC manufacturing techniques can reduce residual vinyl chloride monomer levels to less than 1 ppm, thus minimizing the toxic effects of the compound. Similarly, modern emission-scrubbing equipment can adequately prevent releases of HCl and other effluents during incineration disposal. On the other hand, di-2-ethyl-hexyl phthalate (DEHP) is known to enhance red blood cell (RBC) membrane stability that prevents RBC from rapture.

With regard to the leaching of the plasticizer DEHP, however, expert opinion remains divided. California's Safe Drinking Water and Toxic Enforcement Act of 1986 raised concerns about the toxicity of DEHP. But a long-term hemodialysis study that covered more than 7 billion patient-days of exposure resulted in no widely accepted data linking DEHP exposure to carcinogenicity in human beings.[15]

Advantages of Metallocenes. The revolution in polyolefin materials spurred by new metallocene catalyst technologies has created a great opportunity for medical and health-care industries. High yield, high clarity, high impact resistance, and low extractables are just a few useful characteristics of this plasticizer-free polyolefin family.

Metallocene PP is one group of compounds for which research has shown great potential.[16–17] Unprecedented control over the microstructure of PP has led to commercial production of syndiotactic PP (s-PP); material scientists are also exploring new elastomeric PP using oscillating catalysts.[18] The material properties of these two new PPs are similar to those of thermoplastic elastomers (TPEs), particularly the oscillating catalyst compound, which requires only a propylene monomer. This is different from current commercial PP elastomers that are based on the monomer C3 but have C2 and C4 as comonomers.

Moreover, metallocene-based cyclo-olefin has been tested for medical syringe applications. The cyclo-olefin materials possess a high clarity for solution drug prefilled syringes that is easy for visual inspection.

Metallocene PE (m-PE), on the other hand, has been targeted for use as a film in the medical container and packaging industry.[19–20] Enhanced clarity and reductions in both initial seal temperature and crystallinity certainly create many advantages for the medical industry. Metallocene PE is also expanding into medical packaging applications traditionally dominated by ethylene-propylene-diene monomer and ethylene-propylene rubber.

There are a number of other characteristics that make metallocene-based polyolefins attractive for use in the medical industry. Most important, the TPE-like materials are chemically inert and do not interact with drugs. Their narrow molecular weight distribution (MWD) results in low leaching and extractable levels, and their high thermal stability minimizes the need for stabilizers. The materials accommodate gamma radiation, and impact-resistant s-PP film tolerates steam sterilization. Lastly, the compounds are environmentally sound and can be cleanly incinerated or recycled, thereby reducing disposal costs.

Potential Metallocene Disadvantages. The current formulations of metallocenes have a number of disadvantages that researchers may in time overcome. For instance, concern over metal residues makes researchers' efforts to reduce the use of the cocatalyst methylaluminoxane (MAO) a matter of some urgency. Fortunately, some of newly commercially available m-PE have significantly reduced cocatalyst levels. The usefulness of some formulations may also be limited by processing concerns: Because of its narrow MWD and long cystallization half-time, for instance, s-PP is difficult to process.

Sealing presents another difficulty. Metallocene polyolefins are suitable for heat sealing but not for solvent bonding or RF sealing, which are required steps in assembling medical device kits. Similarly, metallocene PE can not be autoclaved because of its low melting point (T_m). Finally, metallocene technology must still confront the key challenge of cost reduction to meet market constraints. Although their potential is great, metallocene polyolefins will have to overcome a number of challenges before they gain wide acceptance in the medical and health-care industries. These include concerns in the areas of product safety and resin quality, product design and processing, and product performance.

3.4 Manufacturing Process

In the medical industry, it often takes great efforts and long cycle time to qualify new polymeric materials and to validate their downstream manufacturing processes before products reach the market. It is well understood that the quality and cost of the medical products depend heavily on the manufacturing processes. From medical manufacturing viewpoints, it is highly desirable to extend the applications of the approved materials to other developing products. This strategy can be achieved by maximizing material properties through an expanding process window to fulfill product performance requirements. Better quality, high throughput rate, less development time, and cost-effective products can be achieved if the manufacturing processes are optimized. Some examples of manufacturing process related material properties and product performance are discussed next.

4 MANUFACTURING PROCESS ON MATERIALS PROPERTIES

Polymeric materials suitable for medical application have passed toxicology and biological testing. Medical manufacturers always like to extend the approved materials to other product lines if the existing materials have potential to meet the product requirements. Moreover, the physical properties of materials can vary with the manufacturing processing, such as extrusion, injection molding, and compression molding. For example, the optical properties of sheeting materials, such as haze, gloss, yellowness index (YI), and gel imperfection, can be manipulated by extrusion processing conductions. Some examples are discussed next.

4.1 LLDPE Pelletization Processing on Haze, Gloss, and Gel Counts

The pelletizing conditions of linear low density polyethylene (LLDPE) resin and its blown extrusion conditions are examined simultaneously to optimize film properties. Figure 16 shows the effect of pelletization melt temperature on haze and gloss at a constant throughput rate (18 kg/h). No significant change in haze and gloss is observed as the pelletization temperature is below 250°C.

However, the gel level in the blown films is significantly affected by the output rate and melt temperature of the pelletization process. Figure 17 shows the effects of pelletization melt temperature and output rate at the fixed blown temperature of 202°C. Lower pelletization temperature provides lower gel level. This might be related to the dispersion of the higher-molecular-weight species. Uniform dispersion of the high-molecular-weight species might not be achieved if the shear force is too low due to a low melt viscosity at the high pelletizing temperatures.[21] The output rate does not show a consistent trend in the gel level

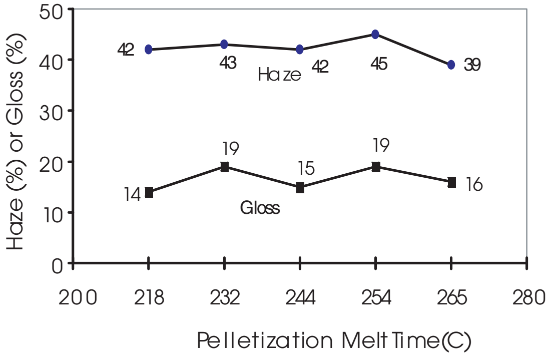


Fig. 16 Haze and gloss of the extruded blown film with different pelletization temperatures. Killion pelletization line was run at 75 rpm and Collin blown line was run at 202°C with 18 kg/h throughput.

of the blown films in Fig. 17. Although, higher shear stresses are provided at higher output rates, high screw speeds could also pass the nondispersed gels through the pelletizing process and result in an initially high gel count in the blown films. The gel level drops dramatically as the pelletization melt temperature decreases to 244–255°C, regardless of the throughput rate of pelletization.

4.2 LLDPE Blown-Film Processing on Mechanical, Haze, Gloss, and Gel Counts

Table 5 shows the data of the selected film properties obtained from the extrusion of LLDPE on the blown-film line at isothermal 190, 218, and 246°C temperature profiles. Ultimate tensile strengths on both machine and transverse directions are reduced at a higher blown temperature. This implies less orientation imparted to the blown films on both machine and transverse directions at a higher blown temperature. The higher blown temperature does essentially no harm to the mechanical properties of the films. The gel level remains at the same level, regardless of change in the blown film melt temperature. From the above study, we conclude that the gel level is only affected by pelletizing temperatures.

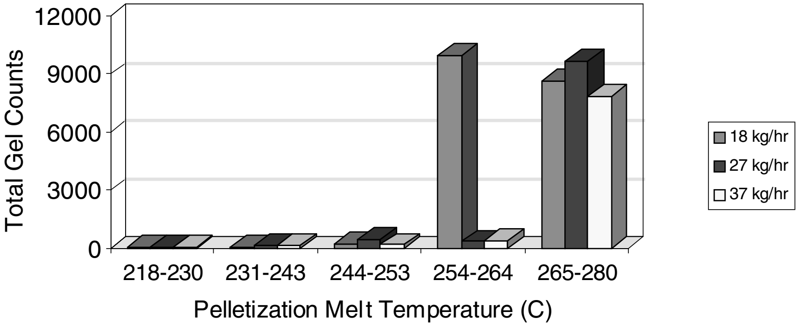


Fig. 17 Total gel count of LLDPE blown films, a different pelletization melt temperature, and different output rates.

Table 5 Effect of Blown Extrusion Temperature on LLDPE Film Properties

Blown Film Temperature Profile	190°C Profile	218°C Profile	246°C Profile
Haze (%)	57.5	45.8	16.8
Gloss (%)	7.6	15.6	62.6
Ultimate tensile strength (MPa) MD/TD	58/50	58/48	42/46
Ultimate tensile elongation (%) MD/TD	902/1002	860/960	840/950
Elemendorf tear (g) MD/TD	3.3/4.8	3.4/4.8	3.5/4.5
Gel counts			
0.2–0.5 mm	68	50	70
0.51–0.7 mm	4	2	2
0.71–1.5 mm	0	0	0
>1.5 mm	0	0	0

MD = machine direction.
TD = traverse direction.

Figure 18 shows the haze of the films processed at different blown-film melt temperatures. The haze decreases from 42 to 17% as the blown temperature increases from 190 to 246°C. Reduction of haze with increasing blowing film temperature can be related to the mechanical deformation of the melt that reduces both melt elasticity and haze. Studies[22] on blown LDPE films suggested that the haze observed in blown-film operations was from melt flow disturbances on the film surfaces at the die exit and crystallization in both surface and bulk. The surface haze moiety or surface roughness from melt flow disturbances could be minimized by reducing melt elasticity through “shear refining” or higher melt temperatures.[23] In Fig. 18, gloss of the blown films increases from 15 to 60% as the blown extrusion temperature increases from 190 to 246°C. Gloss has an inverse relationship with surface roughness.[24] It is believed that the surface roughness of the LLDPE blown films is reduced at a higher blown extrusion temperature just like the responses from the study of low-density polyethelene (LDPE) films.[18] Therefore, an ideal LLDPE film with fewer gels, lower haze, and higher gloss can be obtained by pelletizing the resin at a low-temperature profile, and then blown extruding the pellets at a higher temperature profile. However, the potential challenges could be extrusion stability due to low viscosity from high-temperature extrusions.

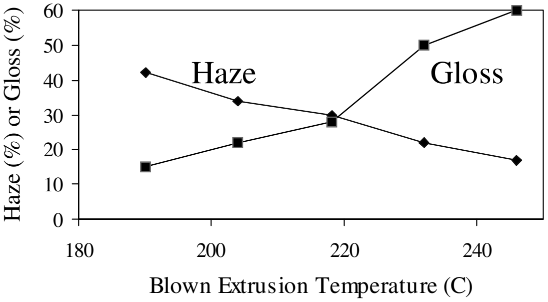


Fig. 18 Haze and gloss on LLDPE blown films with different blown extrusion temperatures.

4.3 Metallocene ULDPE Tubing Extrusion Throughput Rate Improvement

Metallocene polyolefins have narrower molecular weight distribution (MWD) than their corresponding Ziegler–Natta polyolefins due to their catalyst characteristics. Lack of lower-molecular-weight species for a metallocene polyolefin generates manufacturing challenges, such as shark-skin appearance and lower throughput rate. It is desirable to maintain the superior material properties of metallocene polyolefins and improve their extrusion processes by broadening their MWD. Blending a minor amount of a second polyolefin is commonly used to broaden the MWD of a metallocene polyolefin.

As we process the metallocene-ultra low density polyethylene (m-ULDPE) resins, the melt pressures become higher as throughput approachs their maximum at a given processing condition. The melt pressure can be reduced by introducing a thixotropic species, such as a lubricant or the other polyolefins with lower molecular weight. Several secondary polyolefins are blended with the base m-ULDPE to address this challenge. Melt pressure becomes a suitable indicator for throughput enhancement in the tubing extrusion process. A lower melt pressure generally yields a higher extrusion rate.

Figure 19 shows the melt pressure of 1.0 melt index (MI) m-ULDPE tubing extrusion, which gradually drops as the weight percentage of 30 MI m-ULDPE increases. The melt pressure decreases to its original 65% level as the 30 MI resin increases to 25% of the blend. The same resin blend has been run in the commercial-scale extruder and the throughput rate was noted to double that of the base resin. It is believed that 30 MI m-ULLDPE resin enhances the extrusion throughput by broadening the MWD, in addition to its lubricant effect.

The throughput rate increased through the blending approach is also confirmed by the other secondary 18 MI m-ULDPE. Figure 20 shows a similar trend with a slightly less throughput rate increase than 30 MI. This suggests that with similar molecular structure, the secondary resin with a higher MI acts like an effective lubricant that changes the rheological properties of the melt.

Figure 21 shows the extrusion melt pressure of the same base resin that is blended with a 20 MI LDPE resin. A similar trend of extrusion melt pressure is observed as the percent of LDPE increases. The melt pressure of the blend with 25% LDPE decreases to 70% of its original value. LDPE is generally known as a broad MWD resin that is easy for common extrusion processing. It

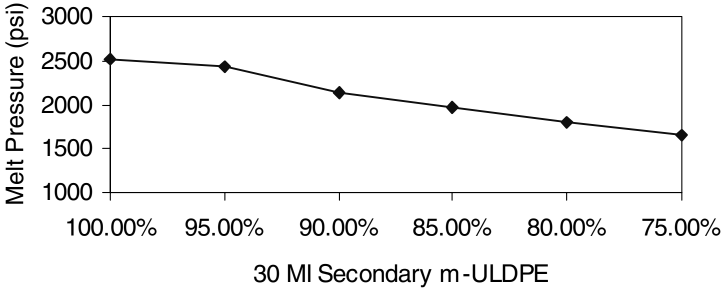


Fig. 19 Melt pressure of 1.0 MI (primary) and 30 MI (secondary) m-ULDPE blends.

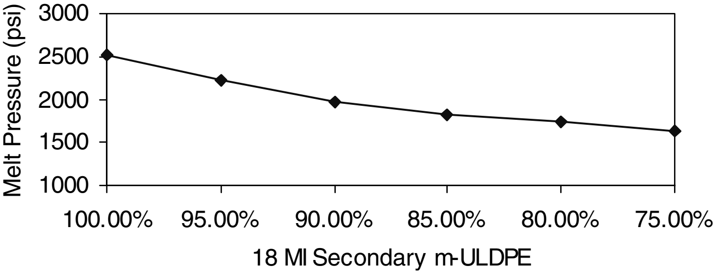


Fig. 20 Melt pressure of 1.0 MI (primary) and 18 MI (secondary) m-ULDPE blends.

is believed that the effect of broadening MWD by LDPE is similar to that by high MI m-ULDPEs.

It is interesting to note in Fig. 22 that a 3.6 MI Ziegler–Natta ULDPE resin does not achieve the similar result. Throughput rate of the blends does not increase even if the composition of the secondary resin increases to 25%. It suggests that broadening the MWD alone is not enough to lower the extrusion melt pressure. The rheological properties of the secondary resin also play major roles in improving the extrusion throughput rate. A typical rheological indicator, such as melt index (MI) number, can be used for this purpose. For the tubing extrusion of 1.0 MI m-ULDPE, an ideal extrusion throughput rate relies on higher MI of the secondary ULDPE or LDPE resins.

4.4 PP Injection-Molding Processing on Medical Product Clarity

The effect of molding temperature on the clarity of medical products made from PP pellets is shown in Fig. 23. Plaques were molded at different temperatures from the pellets having identical melt flow rate and additive packaging, but various catalyst systems. As the injection temperature varied from 184 to 232°C, the YI of the first sample decreased from 10 to 1.0, and that of the second sample from 5.7 to 3.1. This decrease in YI, or improved clarity, that takes place as the temperature increase can be related to resin morphology changes brought on by fast quench from high molding temperatures.[25]

In general, the YI of medical PP suitable for medical product applications is typically expected to be less than 8.0. The variation in YI with molding tem-

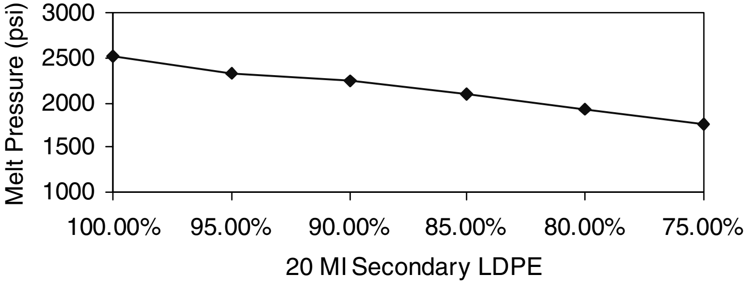


Fig. 21 Melt pressure of 1.0 MI m-ULDPE and 20 MI LDPE blends.

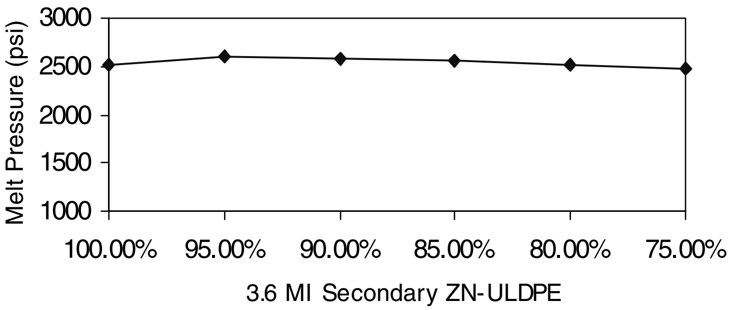


Fig. 22 Melt pressure of 1. O MI m-ULDP and 3. 6 MI ZN ULDPO blends.

perature for different pellets reflects the different catalyst systems used to polymerize the PP flakes. The various levels of catalyst residues introduce different lamellae structures during the cooling process, which in turn results in various degrees of YI reduction.

Figure 24 shows that the YI of plaques molded of PP flakes was essentially independent of the injection-molding temperature. The plaques were molded from flakes that had been pelletized without any additives. Figure 24 indicates that the catalyst residue alone does not cause PP flakes to change in clarity as molding temperature increases. However, the previously mentioned differences in YI between samples when the additives were involved suggest that the metal residues interact with the additives package, and that the effect of their interaction on resin color depends on the molding temperature.

4.5 PP Compression-Molding Processing on Medical Product Clarity

For medical products made by compression molding, temperature again plays a very important role in influencing clarity. Figure 25 shows that a lower YI was observed when medical products were compression molded at 246°C versus at 218°C. In addition, longer compression-molding process produced a lower YI, at both temperatures. PP plaques preheated for 3 min without pressure and followed by 1 or 3 min with pressure generally had higher YI values than those

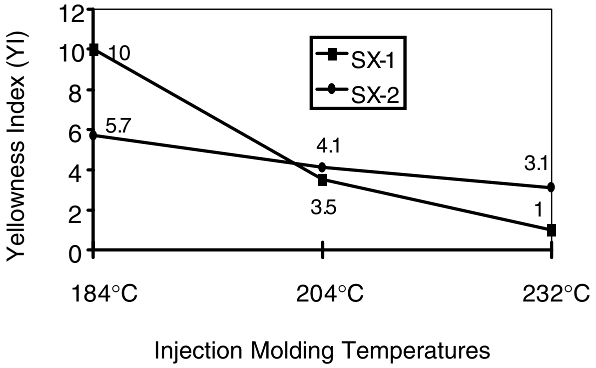


Fig. 23 Clarity of PP pellets improves (YI↓) as injection-molding temperature increases.

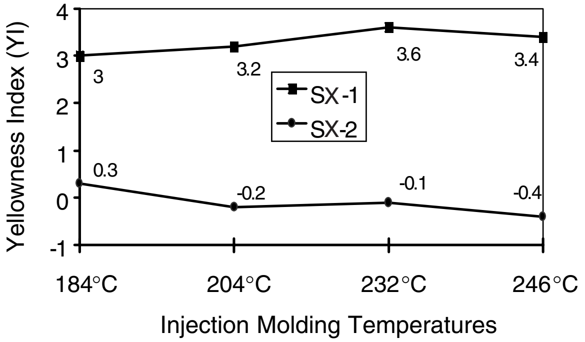


Fig. 24 Clarity of PP flakes is essentially independent of injection-molding temperature.

molded with the corresponding time and pressure continuously applied. The degree of compression pressure, however—observed at either 500 or 1000 psi—has much less influence on the YI of plaque than temperature and molding time. Accordingly, for a given additive packaging, the YI of medical products made from PP pellets can be as high as 10.5 or as low as 0.8, depending on how the product is compression molded.

5 PRODUCT PERFORMANCE

Safety, efficacy, quality, integrity, functionality, and cost are the key factors that determine a medical product's success in a competitive market. A high-quality product performance associated with a low product cost is the ultimate goal of medical manufacturers throughout the world. To achieve this goal, medical manufacturers must carefully select materials to fulfill product safety and efficacy, ensure product integrity, prevent material–drug interactions, maintain strict quality control, and prohibit dose concentration change.

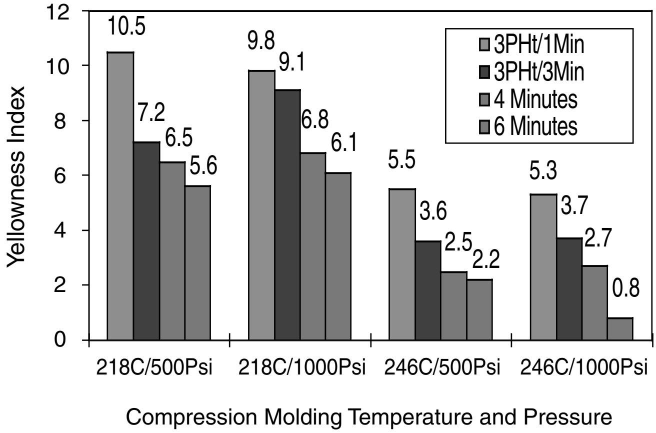


Fig. 25 Compression-molding temperature, pressure, and time on PP clarity (3 PHt/1 min = preheating 3 min then molding for 1 min).

5.1 Correlating Product Performance to Materials Characteristics

Numerous medical products, such as containers and tubing used in the device and medication delivery system, are made from flexible plastics such as plasticized PVC. This is due to its wide formulation latitude, ease of processing, clarity for contamination inspections, and low cost. In many instances these devices and delivery systems are subjected to the simultaneous conditions of subambient temperatures and mechanical stress. For example, near freezing conditions can be expected during winter shipping. Blood components are frequently handled and centrifuge processed at near 4°C. Certain drugs in premixed solutions are often stored and shipped frozen at about −20 to −30°C to enhance the chemical stability. Dry ice storage and transportation calls for temperatures of about −78°C, and in cryo-preservation liquid nitrogen at −196°C is commonly used. Since many products need to be stored in subambient temperatures, low-temperature toughness is needed.[26,27] And yet since many medical products require terminal sterilization by steam, high-temperature survival is a must.

Early studies by Schmieder and Wolf[28] shown in Fig. 26 that glass transitions of plasticized PVC followed a predictable functional relationship with the plasticizer type and content. As expected, the higher content plasticizer samples had lower glass transition temperatures (T_g).

Figure 27 presents the product failure rate in a standardized test conducted at −20°C as a function of the glass transition T_g. Failure rate percent is increased as PVC T_g is higher. This data indicated that the primary variable for the low-temperature performance for flexible PVC is the location of the T_g. This also clearly indicates that the inherent properties of selected materials have a great impact to the product performance.

5.2 Optimizing Product Performance

Product performance can be optimized through the combined synergetic effect of the product design, materials selection, and manufacturing processes. One key element is to define clear and complete product requirements at the very begin-

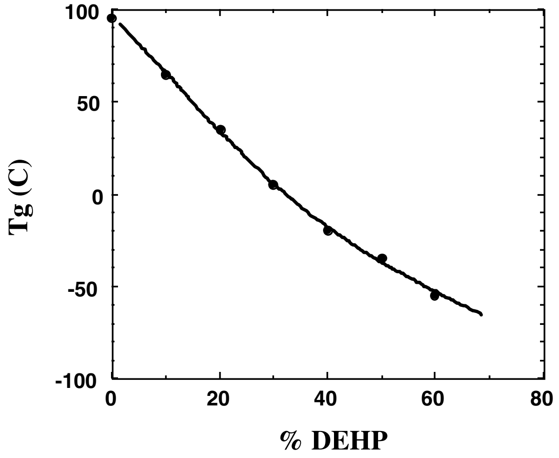


Fig. 26 PVC T_g vs. plasticizer content.

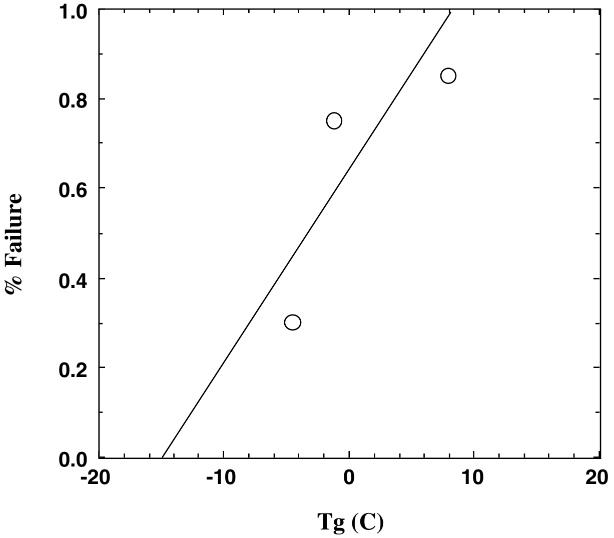


Fig. 27 −20°C product failure rates vs. T_g.

ning of product development. Based on the product requirements, product designers, materials scientists, and manufacturing engineers can create synergy to build a low-cost, high-performance medical product.

6 CONCLUSIONS

Polymeric materials have played a very critical role to enhance medical therapy and product quality, particularly for the medical device and disposable product sectors. Demonstration of product safety and efficacy are required prior to regulatory approval and product launch. Polymeric materials offer flexibility to product design, easy manufacturing, and sterility integrity.

A good materials selection strategy for a specific medical product is to take into account the material functional requirements with product design, manufacturing processes, and product performance simultaneously. The selected materials need to be compatible to the preferred sterilization method of steam autoclaving, ethylene oxide, or ionizing radiation. High-performance low-cost medical products are feasible as the selected materials are suitable for large-scale production by automation processes.

PVC materials have been used for medical products for over 40 years because of broad spectra of physical properties, sterilization compatibility, cost performance ratio, and meeting medical regulatory requirements. Recently, developments in metallocene polyolefins have many unique features that have potential to replace PVC for medical applications.

In addition to product safety and efficacy, the selected materials need to fulfill product functionality, integrity, manufacturability, and cost constraints. Validation of the materials for specific product applications to the desired product shelf is required. The validation includes functionality, stability, and extractives, with focus on the toxicological and biological interactions of the selected polymeric materials with the medication drugs and biological agents.

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CHAPTER 39

MATERIALS IN ELECTRONIC PACKAGING

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1 GENERAL

The process of materials selection for an electronic packaging application involves a variety of considerations, and the end result is often a material chosen for reasons not immediately evident at the beginning of the process. This seeming paradox stems from the nature of the work faced by the electronic packaging engineer. That is, the span of problems faced by the electronic packaging engineer is multidisciplinary and involves finding a balance between function, performance, manufacturability, reliability, and cost.

Handbook of Materials Selection. Edited by Myer Kutz

The focus of this chapter is on narrowing the range of choices regarding selection of the most appropriate solution to a materials selection problem, and this chapter does not set forth a detailed summary of material properties. As a result, the reader is directed to other chapters in this handbook and references cited for specific details regarding the properties of materials, specific manufacturing and assembly processes, and applicable analytical procedures that may apply to the candidate materials.

2 APPROACH

The selection of a material from a set of materials includes the following steps:

1. Define the *dominant* considerations for the design application under consideration.
2. Define any *overriding* considerations that narrow the field of choice.
3. Review *typical applications* regarding materials selection.
4. Compile a list of the most likely set of *candidate materials*.
5. Compare the properties of the selected material(s) and *select one or more materials* that appear to offer the most likely solution.
6. Test each selected material physically and against the original design criteria to *verify suitability*.

3 DOMINANT CONSIDERATIONS

A dominant consideration refers to the primary technical performance requirement that must be offered by the selected material to achieve the technical objectives of the design. The design may also have a set of secondary requirements; however, if the dominant characteristic is not achieved, the material is unsatisfactory for service regardless of whether desirable secondary requirements are present.

It is not unusual for a material requirement to include more than one dominant characteristic: for example, a heat sink material with high thermal conductivity and electrical insulation, or an item to be used in outer space requiring high thermal emissivity and low sublimation, or a component bonded to substrate where the substrate material must have a coefficient of thermal expansion matching that of the component and chemical inertness over a given temperature range.

Dominant technical performance criteria include:

3.1 Electrical Conductivity

A material may be required to conduct electrical currents. This includes metals and some nonmetallic elements such as adhesives, greases, and other compounds loaded with graphite or metal powder. If electrical conductivity is important, then the resistivity of the material must be considered because electrical resistance creates voltage drop and heat generation, either of which may be a desirable or an undesirable consequence. In screening for electrical conductors, a maximum electrical resistance requirement must be defined, thus materials with equal or lower electrical resistivity become candidates for selection. *Electrical resistivity* is temperature sensitive; thus it is necessary to make sure that the material electrical resistivity is satisfactory over the temperature range of interest.

Conversely, it may be necessary to electrically isolate a component or current-carrying member to protect from undesired electrical shorts. Materials used as electrical insulators, such as mica or glass or ceramics and many plastic materials, have very high electrical resistivity, are poor conductors of electricity, and are employed to provide electrical isolation.[1] Some high-voltage dielectrics may be liquid, which often act as heat transfer media in addition to providing electrical insulation. Examples include mineral oils, silicon oils, polybutanes, flourocarbons, and organic esters including castor oil. Problems with liquid dielectrics include limited temperature range, chemical decomposition over time, combustibility, and loss of dielectric strength.

3.2 Thermal Conductivity

All materials conduct heat; however, among materials there is a wide range of *thermal conductivity*. Good conductors of heat include metals, and poor conductors of heat such as ceramic and plastic foam materials are considered to be thermal insulators. The cooling of electronic components and the protection of electronic components from excessive temperatures is managed by employing mounting devices, heat sinks, and thermal insulators using materials of the appropriate thermal conductivity.

3.3 Thermal Emissivity

The *thermal emissivity* of a material is a measure of the efficiency by which a material will radiate or receive infrared energy. The thermal emissivity is a surface characteristic of a material and to some extent a function of material color. Thus a highly polished light-colored material will have a low thermal emissivity and dark-colored, high surface roughness materials will exhibit a higher thermal emissivity.

When an electronic component is mounted near a high-temperature emitter, the heat absorbed by the component may be reduced by using material and finish with a low thermal emissivity to protect the component.

This is of key importance for electronic equipment used in spacecraft and that may face the sun or be in shadow. Low-emissivity materials, such as plating an equipment case with gold, are used to limit the heating from the sun and also to reduce heat loss when the equipment is facing dark space.

If thermal emissivity is a design requirement, often the selected material may be chosen for some other dominant criteria, and thermal emissivity is controlled by surface finish selection, to include application of an electrodeposited highly emissive metal on the exposed surfaces of the part.

Painting a surface with a light-colored paint may slightly lower the emissivity in comparison to darker shades of paint; however, the surface roughness is often essentially the same regardless of paint color, thus the improvement is minor. Also, to coat a dark polished surface with a light-colored paint often leads to a higher emissivity and not a lower emissivity.

3.4 Thermal Expansion

Measured by a property known as the *coefficient of thermal expansion,* this material property allows the electronic packaging engineer to predict the linear or volumetric change (expansion or contraction) of a material when the material is exposed to a change in temperature.

It is often very important to determine the coefficient of thermal expansion for materials that are bonded or mechanically secured together. For example, for a ceramic component (very low coefficient of thermal expansion) soldered to a printed circuit board (a higher coefficient of thermal expansion) exposed to repeated temperature cycles, perhaps caused by turning the equipment on and off, can lead to solder joint failure. Another example is when two materials of different thermal expansion characteristics are mechanically joined and exposed to a temperature change; one material will be placed in compression and the other material will be placed in tension. This will result in the composite assembly bending in the direction of the material placed in tension. Also, depending on the relative strength of the materials, one or the other material may fracture, or the joining method (adhesive joint, solder joint, weld, rivet, screw, etc.) may fracture or otherwise be degraded. Another effect caused by thermal expansion in a circuit where mechanical position defines a circuit operating parameter would be a change in position of parts during a change in temperature, which causes the circuit operating parameters to deviate.

3.5 Chemical Inertness

Materials may be exposed to chemicals during the life of a product. This may include fuels and lubricants, cleaning fluids, fluxes, and other chemicals used in industrial processes. Most tables that list material properties also provide limited information regarding *chemical resistance.* If the material is exposed to conditions not covered by information in the tables, then the designer must contact the provider of the material for additional information. It also may be necessary to experimentally determine and to verify the suitability of a material in the presence of a chemical. It is important to note that chemical reactions are affected by temperature and the presence of other chemicals.

Chemical deterioration may be induced by ionizing radiation (which alters the molecular structure and thus the physical properties of materials) and ultraviolet radiation (such as from sunshine), which may cause depletion of plasticizers from elastomers, like polyvinyl chloride (PVC) plastic, which is used as a wire insulation material, thus rendering the material brittle and weak.

3.6 Corrosion

Most tables of material properties provide information of corrosion resistance; however, this information may not be sufficiently specific to give confidence regarding the intended usage of a material. This may (for metals) include susceptibility to intergranular or stress corrosion cracking in the presence of certain acids or bases. For all materials, and especially nonmetals, corrosion deterioration includes processes similar to those identified regarding the chemical inertness of a material.

The process of galvanic corrosion[2] is a primary concern to the electronic packaging engineer and applies mostly to metals, and under certain conditions galvanic corrosion may be a leading cause for loss of electrical conductivity or loss of strength in a material or at a riveted, bolted, or welded joint as well as failure of electroplated finishes.

Table 1 lists the galvanic series relative position of several materials used in electronic packaging.

Table 1 Galvanic Series Position of Common Electronic Materials

Magnesium and alloys	*Most Anodic*
Zinc	↑
Aluminum 1100 alloys	
Cadmium	
Aluminum 2024 alloys	
Steel, iron, cast irons	
Type 304 and 316 CRES (active)	
Lead–tin solders	
Lead	
Tin	
Nickel (active)	
Inconel (active)	
Brasses, copper, bronze	
Monel and copper nickel alloys	
Monel	
Silver solder	
Nickel (passive)	
Inconel (passive)	
Type 304 and 316 corrosion resistant steel CRES (passive)	
Silver, gold, platinum	
Titanium	↓
Graphite	*Most Cathodic*

Galvanic corrosion is the result of an electrochemical reaction, which occurs when a design that employs metals ranking at different levels on the galvanic chart is in the presence of an electrolyte (i.e., saltwater, contaminated water, or other conducting chemicals). One material acts as an anode and the other as a cathode as in a simple storage cell. The result is ionic migration during which the anodic element suffers sacrificial deterioration. This is a mechanism for leaching elements from grain boundaries and a corresponding weakening of the affected material and other undesirable effects. This process may be hastened or hindered by passing a small electrical current through the affected area.

3.7 Temperature Range

The operating and storage temperature ranges applicable to the application of a material must be known and the properties of the material reviewed for stability over the range of temperatures. This is important for any material including metals especially when the temperatures are very high or very low. Metals and nonmetals will become brittle and stiff at lower temperatures, and may soften, lose strength, and creep or flow at elevated temperatures. This is especially true with nonmetal materials, where the temperature ranges may be severely limited. It is necessary to review the temperature range suitablity of a candidate material because temperature influences each material's physical property differently, even for different alloys or formulations within a given class of materials.

3.8 Strength

A guide to the load-bearing capacity or strength of a material is its *tensile strength*, which for many metals is a function of process conditioning or heat treating (i.e., is the material annealed, half-hard, or hard?). Strength of nonmetals (plastics) varies significantly with temperature and the rate at which a load is applied (impact strength). Weight reduction is achieved in electronics by using smaller quantities of high-strength materials for forming brackets, structures, and fasteners.

3.9 Density

Density is a measure usually taken as the weight of a material for a given unit of volume. This property may be used as a guide to reducing weight in a design. Material density establishes the inertia and resonant frequency of mechanical elements and thus the degree to which they are affected by mechanical vibration and mechanical shock loading.

Another indicator of the density of a material is its *specific gravity*, which is a comparative ratio of the weight of a material compared with the weight of an equivalent volume of water.

3.10 Electromagnetic and Electrostatic Shielding

Electromagnetic shielding is employed to prevent the imposition of electrical waves from the environment (such as radio waves) on circuit elements and thus inducing currents and voltages leading to circuit malperformance and possible component damage.

Electrostatic shielding is employed to prevent penetration of high-voltage charges on the surface of an electronic enclosure (such as caused by static discharge) from entering the enclosure and therein causing damage to components or causing circuit performance malfunction.

Protection of an equipment from both of these sources of potential equipment performance deterioration involve shielding the equipment such as to prevent radio waves or other electromagnetic noise from entering the enclosure, to shield the equipment to promote the collection of static charges on the surface of the equipment, and to dissipate the unwanted energy by providing a conductive path to drain the energy to an appropriate sink (ground).

A circuit may be shielded by enclosing it in a conductive enclosure, thus creating a Faraday cage where the imposing electromagnetic waves or static charges cause currents in the skin of the enclosure and do not penetrate the enclosure. An example would be to protect a high-gain amplifier circuit from malfunction when exposed to radio waves.

Conversely, essentially identical techniques are used to contain radio wave emissions from a circuit from escaping the enclosure and causing malfunction in other equipment that may be susceptible to such emissions. High-speed digital circuits at speeds of 10 kHz and above are known to cause such emissions and often require electromagnetic shielding to meet the emission requirements set forth by Part 15 of the Federal Communications Commission Rules and Regulations.[3] Nonmetallic enclosures are sometimes used to control electromagnetic field by coating the material with a paint that contains metal powder.

The primary guide to the effectiveness of a material as a electrostatic shield is the *electrical conductivity* of the material.

3.11 Magnetic Shielding

Magnetic shielding pertains to protecting a circuit from the presence of fluctuating magnetic field energy that may induce currents in wires and metal structural elements and lead to circuit malfunction or failure. A simple Faraday shield such as used for electromagnetic wave or static discharge protection will not suffice for protection from magnetic fields. It is usually necessary to restrict the shielding materials to high-nickel alloy iron. Iron-bearing alloys are magnetic and block penetration of magnetic flux into an equipment, which may induce noise within a circuit or which may cause malfunction of a circuit including erasure of memory from several classes of memory storage devices such as integrated circuits.

3.12 Fatigue Resistance

Fatigue is the loss of physical strength and eventual failure of a material as a result of the material being subjected to repeated loads. Such loads may be the result of thermal expansion stresses that occur during temperature cycles, mechanical loads induced during the operation of the device, shipping and handling stresses, exposure to vibration, and repeated mechanical impact shock loads.

Repetitive loads are know to cause damage to accumulate until the material fails.[1] Fatigue resistance may be judged on a relative basis by referring to tables of *fatigue resistance*, which relate stress levels to time-to-failure for a specific material or alloy under ideal conditions such as rotating beam test data.

The actual ability of a component to withstand fatigue damage is highly sensitive to the loading history, the precise shape of the component (i.e., stress risers), equipment construction details, and variations in temperature. As a result, proof testing is usually required to verify the ability of the selected material and design details to avoid fatigue damage.

3.13 Hardness

Hardness is taken as the ability of a material to resist denting as caused by a load bearing on the material using a dimensionally standardized probe. There are different scales by which hardness is measured, the most common are the *Brinell hardness* and the *Rockwell hardness number for a given Rockwell scale*. Unless otherwise specified the hardness in a chart refers to the intrinsic hardness of the base material.

Hard surface materials are used to reduce wear in mechanical latches, sliding surfaces, and electrical contacts. The lubricity of polymers such as nylon and Teflon is used to reduce friction in applications such as drawer slides where a plastic material is used to rub against a hard metal surface.

The hardness of a material is often established by the surface treatment of the material and is not always a bulk property of the base material. That is, steel and aluminum and many other materials may be chemically or physically surface hardened to improve the wear of the part under design. The ability of a material to be surface hardened varies widely among materials, with many materials lacking the ability to be surface hardened.

3.14 Ductility

The *ductility* of a material refers to the ability of the material to be deformed without breaking. Extreme examples would include tin (very ductile) and glass (brittle and thus not ductile). This is an important property for materials that are to be mechanically formed, are subjected to bending stresses during use, and must withstand impact shock loading without cracking or shattering.

A guide to ductility of a material is its *elongation*, which refers to the ability of the material to deform under load before failing. Elongation is seen to vary with the degree to which a material is heat treated (in the case of copper and iron alloys). Ductility in plastic materials is strongly affected by temperature. For example, a plastic gear may work well at room temperature; however, at low temperatures it may become brittle and fracture under load. Material ductility varies widely among even a given general class of materials and each individual material must be reviewed for ductility.

Closely related to ductility is the material's *malleability*, which is the ability of the material (usually metals) to be shaped by stamping, spinning, cold heading, upsetting, rolling, or other physical processes involving deformation of the basic material in solid form. A malleable material is one that may be physically formed without tearing, cracking, fracturing, or developing surface defects. Tables of materials properties often provide guidance relative to malleability, and subcontractors who provide metal shaping services are a good source of information on malleability.

3.15 Wear Resistance

Wear resistance is a property of materials that does not often appear in tables of materials properties. A guide to wear is *hardness*—the harder and smoother the material the greater it's resistance to wear. Also consider ductility for the reason that a material may become brittle at low temperatures and exhibit surface fractures and fretting under load. Wear life is also a function of the magnitude of load-induced localized compressive stresses and the methods used to reduce friction such as lubrication. Some nonmetallic materials, such as nylon, are self-lubricating and useful as bearing materials.

3.16 Sublimation

Sublimation is the loss of material when the material is exposed to low pressures (vacuum) and or elevated temperatures. It involves the conversion from solid form immediately to gaseous form apparently without passing through a liquid phase. Sublimation conversion may occur uniformly to the surface of a uniform and pure material, or if the material is a more complex matrix one or more constituent materials may sublimate leaving a residual material possessing poor mechanical or electrical properties.

Sublimation may also result in the redeposition of material onto another surface at a lower temperature where the presence of the sublimated material is not desired (i.e., perhaps forming a conductive film causing circuit elements to short or reducing visibility through a window). Information on sublimation is limited and may require inquiry to the manufacturer of a material or to experimental test data.

Sublimation is of critical importance when the electronic packaging engineer selects materials that will be subjected to low-pressure and vacuum environments such as on electronic equipment for use in space vehicles.

3.17 Combustibility

Combustibility is the property of a material, under a given set of conditions, to rapidly oxidize or burn, which may range from the ability to burn but not sustain flame when the ignition source is removed, or to burn and sustain flame after the ignition source is removed. Combustion may lead to the generation of noxious gasses and destructive deterioration of the circuit elements in the vicinity of the combustion.

This property is most often associated with plastic and organic materials; however, some metals will burn in the presence of oxidizing gasses or chemicals. Many tables of plastic material properties do not include combustibility information; however, this information is usually available from the manufacturer of the material. If combustibility is to be prevented in a design, it is sometimes necessary to conduct tests to determine if a potential material is suitable for the intended application.

3.18 Creep

Creep is the ongoing deformation (flow) of a material under mechanical load. Failure due to creep is often very slow, and it may occur only during certain conditions (such as a solder-sealed case that is sealed at sea level but operated in an aircraft, or the same case operating at seal level but within which the internal pressure is increased due to heating during normal operation of the equipment). It is good design practice to never place such a soldered joint in sustained mechanical stress for the reason that the solder will creep until the stress is relieved or the joint fails. Thus the design of a mechanical joint where solder is employed (e.g., to achieve an enduring electrical connection or to seal a joint) must have the mechanical load taken by structural members without the solder present. Mechanical joints or seals that utilize adhesives, elastomers, and other nonmetallic materials must be treated in the same way to avoid eventual failure.

A creep failure may not become evident for days, weeks, or even years; but it will happen if materials with the known ability to creep are improperly incorporated into a design.

Creep is not only a characteristic of some metals, it also is present in several inorganic or organic materials as well. An example would the loosening of a mechanical joint where the joint consists of metallic elements bolted together and includes a nylon washer. This joint eventually loosens when the nylon washer creeps to relieve the initial clamping stresses. Another example would be when a compressible gasket, made of a material that will creep, is used in a flanged joint where the joint does not have mechanical stops to control the separation of the flanges when the bolts are tightened. In this case the joint will eventually suffer loosening and possible leakage when the gasket creeps to relieve the joint compressive forces. A classic example of a creep-induced failure occurs when an insulated wire presses against a hard metallic object such as the edge of a bracket or panel. In this case, the insulation will creep until the inner conductor of the wire eventually makes contact (shorts to) the metal object.

3.19 Moisture Absorption

In many instances when a material is selected for use as an insulator, or the material must have dimensional stability in humid environments, the *moisture absorption* characteristics of the material becomes important. For metals, moisture absorption is not a significant consideration; however, the presence of moisture will hasten galvanic corrosion when different metals are in intimate contact.

For nonmetals, porous sintered metal structures, and ceramics, moisture absorption is a significant consideration because for an electrical insulator, the insulation capability is reduced due to the absorption of moisture, and the insulator may become electrically conductive to various degrees when moist. Hydrophobic (water-resisting materials, the surface tension of which causes water to bead and not wet the surface of the material) materials are preferred for insulator applications.

Most nonmetallic materials are tested for moisture absorption, and the results of this data are found in many listings of material properties. The majority of moisture absorption data relates the absorption of water over a specified period of time.

In some cases, particularly for organic materials, moisture absorption leads to a loss of strength and physical deterioration. If some materials become wet and are subsequently allowed to dry, they may exhibit permanent shrinkage and the inability to redevelop their original physical characteristics.

Should a material absorb moisture and be subjected to freezing temperatures, the material may be permanently damaged due to expansion that occurs upon freezing.

The chemical absorption properties of seals, paints, tubing, gasket materials, and plastic materials expected to be used in applications involving chemicals (oil, gasoline, fuels, alcohol, cleaning solutions, etc.) most likely have been investigated, and the materials supplier is usually able to provide test data and guidance in the selection of an appropriate material.

Also, in conditions of high humidity and moisture in the presence of inorganic salts, conditions are created that encourage the growth of fungus. Fungus growth can lead to material degradation in appearance and loss of strength and the development of undesirable electrical shorts. Materials are generally categorized as fungus nutrient or fungus resistant. Materials such as metals, glass, ceramics, mica, silicone resins, asbestos, acrylics, diallyl phthalate, and a limited list of plastic resins are fungus resistant. Materials containing organic constituents, rubber, cellulose acetates, epoxy resins, many lubricants, medium- and low-density polyethylene, polyvinyl compounds, formaldehyde compounds, and others are fungus nutrient. The above listing is far from complete, and the designer is cautioned to verify the fungus nutrient characteristics of any materials used in a design.

4 OVERRIDING CONSIDERATIONS

It is not unusual for an electronics packaging engineer to find that there are overriding considerations that impact the materials selection decision. Often there is little if any opportunity to change the overriding considerations, thus the materials selection must be made in a manner to best achieve the dominant technical performance requirements within a framework of overriding considerations.

Overriding considerations may include:

- Customer preference for a given shape, material, or finish
- Ergonometric (human factors) considerations
- Product is part of a family of products that must have identical materials
- Product manufacturing technique limited to available factory machinery
- Industrial design constraints dictated by the application
- Part to be designed must interface with a component or material for which there is no alternate
- Service temperature range
- Shock or vibration environment
- Physical abuse in service
- Exposure to chemicals
- Mechanical strength
- Exposure to electromagnetic, electrostatic, ionizing, ultraviolet, or infrared radiation
- Corrosive environments
- Availability of the material

5 TYPICAL APPLICATIONS

An important guide to materials selection is found by considering how materials have been used for like or near-like applications. Electronic packaging applications often fall into one or more of the following applications:

- Equipment attachment
- Equipment racks, frames, and mounting structures
- Equipment and module enclosures
- Temperature control
- Mechanical joints
- Finishes
- Position-sensitive assemblies
- Electrical contacts
- Harsh environment endurance

5.1 Equipment Attachment

Other than desktop and portable equipment, electronic equipment is usually mechanically fastened to an equipment rack, frame, shelf, or mounting structure. These devices may sit on the floor in an equipment room or are bolted [using high-strength Society of Automotive Engineers (SAE) rated fasteners) or welded to the structure of the containing building or vehicle. The attachment may consist of, for relay rack-mounted devices, passing two or more bolts through holes in the equipment front panel into threaded fasteners in the rack frame. For equipment used in aircraft, ships, and vehicles, ARINC standard modular ATR cases are placed on an equipment self and secured thereto. The ATR module securement usually consists of an engagement device in the rear of the module and brackets with retained thumbscrews or latching hook arrangements at the lower edge at the front of the module, including a device fastened to the shelf into which the screws or latches engage. Larger equipment used in moving vehicles, such as tanks, battleships, and trucks, where the equipment must be protected from higher levels of vibration and shock loading, often involves bolting the equipment to a frame, which incorporates energy-absorbing dampers, and bolting the frame to the structure of the vehicle. For consumer goods applications (car radios and CD players, motorcycle and bicycle electronics, etc.), a variety of mounting configurations are employed; some feature quick release of the attached equipment.

Each of the applications place similar requirements on the materials used for attachment. The dominant considerations are strength, wear resistance, corrosion avoidance, temperature range, and fatigue resistance. The materials are usually selected from the steel family to achieve a favorable strength-to-weight ratio. For mounting bolts used to fasten commercial goods and other equipment used in relatively benign environments, cadmium- or nickel-plated steel hardware is used. If the mechanical loads are severe, higher strength SAE-grade steels must be used. For more corrosive environments, stainless steel (usually passivated) is employed. For aggressive high-stress applications where corrosion and wear are a concern, fastening devices and brackets are made from the precipitation-hardened stainless steels that are passivated.

5.2 Equipment Racks, Frames, and Mounting Structures

Equipment racks, frames, and mounting structures such as an equipment shelf are designed to bear the weight of electronic equipment mounted thereto. The dominant considerations are strength, wear resistance, corrosion, electrical conductivity, and corrosion avoidance. The materials of preference include heat-treated high-strength aluminum (such as 6061-T6) and mild steel materials. The aluminum materials are almost always either anodized or treated (chromate dip or similar) and painted for appearance and protection from the elements. Steel materials are protected from rust by nickel, zinc, or other electroplated finish and often by a rust-resistant paint. Stainless steel parts are passivated to improve resistance to corrosion.

5.3 Equipment and Module Enclosures

Enclosures are the cases or boxes that house electronic equipment. Dominant considerations include operating temperature range, electromagnetic and electrostatic shielding, magnetic shielding, strength, wear resistance, thermal conductivity, appearance, corrosion resistance, and perhaps chemical inertness.

Materials used for enclosures vary with the application and end-user market. They include both metal and plastic materials. Cost containment, ease of fabrication, and appearance are often the overriding considerations.

When electromagnetic, electrostatic, and magnetic shielding protection is not required, plastics materials are often used to fabricate enclosures. Aluminum and steel materials may be utilized to achieve electrostatic and magnetic shielding protection. If magnetic shielding protection is required, iron-based materials are required, and the material of choice is usually steel. Metal cases are almost always protected against corrosion and other environmental damage by a finish treatment, sometimes followed by paint to achieve a suitable appearance.

Plastics materials may be used for cases for electromagnetic and electrostatic protecting enclosures if they are coated internally with a conductive paint that may bear either silver or nickel powder. Enclosures fabricated from plastic usually do not require application of paint, chemical treatment, or other surface protection. Another dominant consideration for plastic cases is combustibility for the reason that many plastics will burn and emit noxious or toxic fumes if exposed to flame or high temperatures.

5.4 Temperature Control

Temperature control is directed at maintaining electronic component temperatures within prescribed limits. This is accomplished by the proper selection and application of materials used to conduct heat from or to the components.

The primary modes of heat transfer are conduction, convection, and radiation. The dominant considerations for heat transfer material selection include thermal conductivity, chemical inertness, resistance to corrosion, and sometimes wear resistance, sublimation, thermal expansion, and the ability to resist ionizing and ultraviolet radiation.

Conduction heat transfer involves the flow of thermal energy through a material from a heat source (component) to a heat sink. The design goal is to reduce the resistance to heat flow between the heat source and the heat sink. The effectiveness of heat transfer within a material is measured by its thermal conductivity. Metals such as silver, copper, and aluminum are good heat conductors, however, aluminum is most often used because it tends not to tarnish, is easy to form, and costs less. Copper or copper alloys are used when large amounts of heat are to be transferred and any small improvement in thermal conductivity is meaningful in the design.

When the design requires electrical insulation and good thermal conduction across a joint between two materials, thin layers of mica, silicon-impregnated cloth, or ceramic materials are often used. These joints may be enhanced by applying a thermal grease to the joining parts to prevent the entrapment of air or other gas (a thermal insulator) between the mating surfaces.

Convection heat transfer involves the movement of heat between one material (usually metal) and a surrounding material such as a gas (air) or a liquid (water, freon, etc.). The usual design seeks to conduct heat from a heat source into a physical configuration, such as fins, which present a relatively large area of contact between the metal object and the gas or liquid with which the heat is to be exchanged. Good thermal conductivity is very important, however, so is strength and resistance to corrosion. Convection heat sinks are often made from the high-strength aluminum alloys to meet these requirements. For resistance to corrosion, which may result in the formation of a thermally insulating film between the heat fin surface and the heat transfer media, the aluminum heat sink is sulfuric acid anodized black in color to also enhance heat transfer by radiation. Painted surfaces are avoided because paint acts as an insulator and reduces the efficiency of the heat transfer surface.

Radiation heat transfer is the primary mode of heat transfer when considering space applications. Radiation at sea level can contribute 10% or more of the total heat transfer process depending on the temperature gradient and distance between the radiating and receiving objects. Radiation heat transfer applications feature use of thermally conductive materials to transfer heat to radiating surfaces. The effectiveness of the radiation surfaces is dependent on the finish applied to those surfaces. For materials that are to reduce the reception or reduce the ability to emit heat, the surfaces are smooth and shiny. Such surfaces are often electroplated with a material such as gold, chromium, or nickel, which are chemically inert and may be buffed to a very high gloss. For materials for which the design requires effective heat absorption or emission, the material surface is not smooth (thus to increase surface area) and is usually black or another dark color. This is achieved on aluminum surfaces by application of a black sulfuric anodize finish such as used on the internal elements of solar collector devices.

Paint on a surface adds a layer of insulation and thus is an impediment to radiation heat transfer. Due to the surface roughness of painted surfaces, the color of the paint usually makes only small differences in surface radiation effectiveness; however, a light-colored (white) paint will be somewhat less efficient at radiation heat transfer than a dark (black) paint under extreme thermal loads.

Plastic and ceramic materials are not effective radiation heat transfer materials and may be employed as surface insulators to reduce the effectiveness of radiation heat transfer.

5.5 Mechanical Joints

Mechanical joints are structural attachments within an electronic rack, enclosure, case, shelf, or housing and are materials sensitive. Mechanical joints may be either permanent or semipermanent. A permanent mechanical joint could be brazed, welded, adhesively bonded, chemically joined, riveted, or mechanically upset. Semipermanent mechanical joints involve the application of mechanical fasteners and permit the mechanical joint to be attached and detached during service.

Mechanical joints may involve two or more metal elements, two or more nonmetallic elements, or a mixture of both metallic and nonmetallic elements.

For metallic-only fused joints the metals must be suitable to the joining method and carefully selected filler materials achieve the joint, such as welding rod alloy materials or brazing materials. The dominant considerations include temperature range (will the material properties be altered by the temperatures required by the fusing process?), relative thermal expansion to avoid residual stresses, and avoidance of materials that in combination will lead to galvanic corrosion. If the metals gain their strength properties by being heat treated after forming, the joint materials may require annealing prior to fusing and heat treating after fusing.

Soldering metal parts together using conventional electronic lead–tin solders is not a valid method by which to form a mechanical joint. Solder will creep under mechanical load and if the mechanical load is continuously applied, the soldered joint will eventually creep to failure. Solder is a valid method for electrically bonding and environmentally sealing mechanical joints, such as riveted joints, which are designed to carry mechanical stress without the presence of the solder and where the solder is used for bonding or sealing and is not used to carry mechanical loads.

Nonmetallic (plastic) material joints may be formed by thermal bonding or ultrasonically welding identical formulations together (thermoplastics only) or by chemical fusing agents as recommended by the plastics manufacturer.

Mechanical joints involving a variety of materials (i.e., both metals and plastics) require careful materials selection, and the dominant characteristics of interest include thermal expansion, corrosion, creep, temperature range, and electrical conductivity.

5.6 Finishes

The majority of metallic materials and many plastic materials require the application of a surface finish to enhance resistance to corrosion and to provide an attractive appearance. Dominant considerations regarding application of material finishes include electrical conductivity, thermal conductivity, thermal expansion, chemical inertness, temperature range, wear resistance, sublimation, and moisture absorption.

Aluminum materials used inside of an equipment are usually chemically cleaned and chromate conversion coated. Chromate conversion coating is also used as a surface treatment that promotes improved adhesion of paint to the aluminum. A chemical-film-coated aluminum passes electrical signals and may have an electrical conductivity greater that the base aluminum. These coatings are easily scratched and have poor abrasion resistance.

Anodizing of aluminum creates a wear-resistant and sometimes attractive finish that consists of a thin layer of aluminum oxide. This oxide film is an electrical insulator and may cause a problem if the part is to pass electricity from to another component. Sulfuric acid anodize is used to provide protection from corrosion, causes very little thickness growth to the base metal, works well for dyed applications, and is avoided if the design has overlapping surfaces where the acid may be trapped during the anodize process. Chromic acid anodize is preferred when small dimensional change is necessary for highly stressed parts or on assemblies that could entrap processing solution. Hard anodize provides and extremely hard finish with excellent abrasion resistance and poor electrical conductivity. Hard anodize coating is brittle and will crack should the anodized part be flexed. In addition, hard anodize causes a loss of strength because for 0.04 mm of film thickness, 0.02 mm of the underlying material is lost. In addition, the anodize thickness increases the part dimensions by about one-half the thickness of the film.

Aluminum may be electroplated to achieve a variety of properties, such as plating with a thin layer of gold to achieve a low thermal emissivity, or selective plating with tin over electroless copper to permit the solder attachment of an electrical conductor to the aluminum part. The entire part should be plated for maximum protection, and voids in the plating may establish conditions for galvanic corrosion.

Steel parts must be protected from oxidizing (rusting) in the presence of even small amounts of moisture or humidity. Steel is usually protected by plating with nickel or zinc for fasteners, and with tin when used as an electronic equipment chassis to which electrical connections are required. Nickel and chrome plating are used on steel parts to improve resistance to wear and to provide an attractive appearance. Cadmium plating is used extensively to protect steel against corrosion; however, it is limited to use below about 230°C and is known to sublimate under vacuum conditions. Other treatments such as black oxide coating and phosphate coating offer alternative approaches to protection of steel surfaces.

Caution is required when a metal is electroplated with another metal. The two materials differ in coefficient of thermal expansion, which may promote delamination of the plating from the base metal when the composite is subjected to thermal cycles. Also, the further apart the materials are listed on the galvanic series, the greater the opportunity for galvanic corrosion to occur. This latter consideration will require the addition of protective coatings (paint, adhesives, etc.) at any location where the plating is removed and both the plating and the base material are exposed to the environment. Aggressive mechanical fastening techniques, such as the use of star-type (shakeproof) lockwashers in a bolted joint, may cause local failure of the plating and eventually lead to joint failure (a loss of electrical continuity, mechanical strength, or both) by corrosion.

Stainless steel parts used in electronic applications are passivated to remove surface contamination and to form a uniform protective oxide film that assures that the material delivers maximum resistance to environmental contaminates.

Other commonly used metals, such as beryllium copper, bronze, and brass also must be protected and may be treated by processes similar to those used to protect steel.

Plastic materials may require finish treatments such as electroplating to achieve surface electrical conductivity or a metallic finish for appearance purposes such as applying chrome on a knob or bezel or other like application. Not all plastics may be painted due to the inability of commonly used paints to adhere to the plastic surface. The surface texture of thermoplastic parts may be given an attractive matte finish by vapor blasting.

5.7 Position-Sensitive Assemblies

Position-sensitive assemblies include devices the operation of which requires an invariant physical relationship between components over the range of temperatures to which the electronic product will be exposed. One example would be an antenna coupler or filter wherein the resonance frequency of the antenna coupler is determined by the length of protrusion of a cylinder (tuning element) into the cavity of the coupler. The protrusion distance is varied by the equipment operator to be proper for each specific operational frequency and once set must be maintained within less than a millimeter over a significant temperature range. A second example would be a variable-frequency tank circuit that after being adjusted must maintain precise values of inductance, capacitance, and resistance over a range of temperature. A third example would be an electronic structure featuring panels or structural elements fabricated from different materials but attached together and must not be allowed to deform when subjected to a change in temperature.

The dominant consideration for position-sensitive assemblies is the thermal expansion of the materials involved. Some materials, such as Invar, a nickel–steel alloy, and structural ceramics have a very low coefficient of thermal expansion and are used to facilitate the design of critical positioning mechanisms.

5.8 Electrical Contacts

Electrical contacts in electronic applications usually consist of switch contacts and connector contacts. The requirement for each are similar; however, the usage varies in that the switch contacts may be frequently operated and the connector contacts are rarely engaged or disengaged.

Materials selected to make electrical contacts depend substantially upon the amount of electrical energy that the contacts must carry. The dominant considerations for selecting contact materials include electrical conductivity, chemical inertness, resistance to corrosion, thermal conductivity, and wear resistance. Electrical contacts are usually made from metal materials; however, in unusual instances where a low electrical conductivity is not required, conductive plastics (plastics loaded with carbon or metallic particles) are employed. Sometimes a metal contact will ride upon a nonmetallic contact such as in a carbon composition potentiometer.

Connectors that must maintain reliable electrical contact over extended periods of time are often designed to produce high contact forces between the contacting elements. High contact forces result in significant mechanical erosion of the contacting materials whenever the connector is engaged or disengaged and can seriously shorten the number of uses of the connector. Connector engagement is beneficial because the mechanical action of the contacting materials sliding over each other tends to remove surface films and corrosion and thus lead to a lower contact resistance. This lowered contact resistance will last until the contact resistance is increased due to the formation of chemical films or corrosion products.

Contacts that are required to carry (make and break) very low electrical current, such as CMOS level signals, are known as “dry contacts.” Dry contacts are fabricated using inert metals or metals plated with an inert metal such as gold or rhodium, upon which surface film formation is inhibited. Silver contacts and lead-based metal contacts are not used because they tarnish, and the tarnish film is a poor conductor of electricity and thus induces resistance in the switched circuit. Long-term resistance to environmental chemicals is sometimes improved by coating the contacts with a protective grease or other media.

Circuit contacts where the circuit carries higher levels of current provide greater freedom in selection of contact materials because the current flow is adequate to dispel the tarnish and other films that may form on the contacts. These contacts are often made from beryllium copper, silver alloys, lead–tin alloys, nickel, rhodium, and other materials.

Dominant considerations for connectors include electrical conductivity, chemical inertness, prevention of galvanic corrosion, hardness, and wear resistance. Many of the same materials used for switch contacts are used for connector elements, however, with more emphasis on wear resistance and chemical resistance. It is necessary to prove electrical contact performance for both switches or connectors by extensive life testing with the primary variables being contact force and material hardness.

5.9 Encapsulation

Encapsulation or potting of an electronic component or subassembly is utilized to promote ruggedness, resist harsh environments, provide electrical isolation, and to promote improved heat transfer for cooling of the encapsulated components. Encapsulation is also used to seal an assembly, thus to protect proprietary design details from the curious.

There are a variety of material properties that must be considered when selecting an encapsulant. Sometimes the requirements are inconsistent with the range of properties offered by any one material. Of interest are the ability of the material to flow freely, not capture or entrain air bubbles, be light weight, offer acceptable electrical, thermal, and chemical properties, and resist cutting and abrasion after cure. During cure the material should not produce excessive temperatures nor produce excessive stresses on the encapsulated parts. After cure, the material should not absorb moisture, not produce excessive forces during temperature cycles, must be stiff enough to support components and connections, and should be able to be removed for purposes of repair.

Some of the properties of the materials exist for a short period of time, such as during cure and for some duration following cure. An example would be short-term dielectric strength that may not stabilize for several days after cure.

There are at least three primary groups of material formulations used for encapsulation:

- *Epoxy Resin–Based Materials.* These materials may be formulated for strength and rigidity. The addition of glass microspheres would reduce density, and addition of metal or other powders will increase thermal and electrical conductivity. Epoxy resins are usually two-part mixtures and require strict control of the mixing, pouring, and curing processes.
- *Silicon-Based Materials.* These materials are know for having an extended temperature range over which they do not degrade. They tend to remain flexible and are resistant to water. They are either one-part or two-part formulations. Some formulations generate acetic acid during cure and may cause corrosion of parts and conductors. Cure time is a function of the thickness and mass of the encapsulant and may require more than one day to achieve full cure.
- *Polyurethane-Based Materials.* These materials generally consist of a catalyst and resin and require mixing prior to pouring. They tend to remain flexible over a useful temperature range and after cure are of a rubberlike consistency; however, they have less tendency to adhere to component, wires entering the potted volume, and potting box surfaces than epoxy or silicone encapsulants.

Epoxy polymers are used to provide low-cost, high-performance reliability without hermeticity to provide environmental protection for bare circuit chips when used in chip-on-board (COB), ball grid array (BGA), multichip module (MCM), and chip-scale packaging (CSP) assemblies.

Data provided on encapsulants is found on manufacturer's data sheets; however, this information may be incomplete and sometimes does not relate physical properties to the method of cure and the material thickness. It is often necessary to consult with the technical support personnel of the manufacturer and to perform testing to determine suitability for the intended design goals.

5.10 Harsh Environment Endurance

Environmental endurance is the ability of the selected material(s) to resist degradation when subjected to the service conditions faced by the equipment or device that is constructed from those materials. Unusually harsh operating conditions may cause the materials selection process to focus on the ability of the material to resist the service environment before the designer considers other important desired material properties.

Applications involving high temperatures require the use of metals, ceramics, and a few plastics.

A highly corrosive chemical environment possibly involving strong acids or strong alkali vapors or solutions are resisted by some glasses, some ceramics, stainless steels, and some plastics.

Solar radiation creates heating of materials and degrades some plastics, such as nylon and PVC, which lose weight and strength when exposed to ultraviolet radiation.

Glasses, ceramics, and many metals react well in the low pressures and the vacuum conditions of outerspace without sublimating or losing strength due to ultraviolet radiation exposure.

6 CANDIDATE MATERIALS

6.1 General

With the dominant and overriding design considerations defined, the electronics packaging engineer is ready to survey general classes of materials for the purpose of identifying one or more candidate material for the design task under consideration.

One primary materials selection consideration is often the conductivity of the material; that is, whether the material is a conductor or an insulator of electrical signals and currents. Conductivity is an important characteristic (printed circuit traces, wires, etc.) and is often the reason that metals are the materials of choice. Other important characteristics of metals include strength, durability, thermal conductivity, and ability to work over a wide temperature range.

Nonmetallic elements may exhibit many of the characteristics of metals; however, they are used to provide electrical and thermal insulation. Other properties such as inertness to chemicals, dielectric strength, appearance, low cost, and east of fabrication may lead to the use of nonmetallic elements.

Semiconductor materials may be employed to achieve, when properly formulated as in transistor junctions, the ability to become either an electrical conductor or an electrical insulator depending on the conditions of use.

Several major categories of materials exist[4] from which the designer may identify subsets and specific materials for detailed review and selection.

6.2 Metals

A guide to the electrical conductivity of a metal is its electrical resistance.[5] Table 2 gives volume resistivities of selected materials, where:

Table 2 Materials Categorized by Resistivity

Conductors	Semiconductors	Insulators
Basic metals	Silicon	Ceramics
Metal alloys	Germanium	Elastomers
Metal bearing compounds	Gallium arsenide	Gasses
Plasma	Selenium	Glass
Some liquids		Solvents
		Thermoplastics
		Thermosets

- Insulators are considered to have a volume resistivity of 10^6 Ω-cm or greater;
- Semiconductors in the range of 10^6 Ω-cm to about 10^{-3} Ω-cm
- Conductors are materials with volume resistivity of about 10^{-3} or lower

Other selected properties of metals used in electronic packaging are provided by Table 3.

Iron and Its Alloys

Iron-bearing materials used in electronic packaging include various alloys of steel.[6] Steel materials[7] are strong and durable; however, they are subject to deterioration by oxidation (rust) and relative to other metals, such as copper and aluminum, are less efficient conductors of electricity and heat.

Steel alloys containing chromium, commonly referred to as "stainless steels" or CRES (corrosion-resistant steel),[8] resist oxidation and tarnishing and are often used in electronic packaging as fastener devices such as screws, nuts, washers, and other hardware. Some alloys of stainless steel are not magnetic, a property that is sometimes useful when used in conjunction with circuits that are sensitive to magnetic fields.

There are a wide variety of properties that may be developed for steel alloys such as very high strength-to-weight ratios, hardness, wear, selective resistance to chemicals, the ability to be formed by mechanical cutting and shaping, and the ability to be joined by welding and brazing. Consumer goods electronic equipment chassis are sometimes constructed from tin-plated steel, which is a low-cost material that can be easily cut or formed and that may be soldered using conventional lead-tin electronic solders.

Aluminum and Its Alloys

Aluminum alloys[9] are commonly used in electronic equipment for manufacturing cases, chassis, covers, brackets, and other mechanical parts. Aluminum alloys range from being "dead soft" through alloys for which substantial strength and surface hardness improvements are achieved by heat treatment.

Aluminum alloys are subject to degradation by both acids and bases and are subject to galvanic corrosion when in contact with other metals. Aluminum is a good conductor of both electricity and heat. Common surface protection methods include iridite or chromate dip coating, anodizing, and painting.

Table 3 Typical Properties of Selected Metals

Metal	Melt Point (°C)	Density $\times 10^{-3}$ kg/m^3 (20°C)	Resistivity $\times 10^8$ Ω-m (20°C)	Thermal Cond. W/m-K (0–100°C)	Thermal Expansion $\times 10^6$ 1/°C
Aluminum	660	2.7	2.7	238	23.5
Antimony	630	6.7	42.0	17.6	8.11
Beryllium	1284	1.8	5.0	167	12.0
Bismuth	271	9.8	116	7.9	13.4
Cadmium	321	8.6	7.4	92.0	31.0
Chromium	1875	7.2	13.0	69.0	6.5
Copper	1491	8.9	1.7	393	17.0
Germanium	937	5.3	$>10^6$	59.0	5.8
Gold	1064	19.3	2.3	263	5.8
Indium	156	7.3	9.1	81.9	25.0
Iron	1525	7.9	9.7	71.0	6.8
Lead	328	11.7	21.0	34.3	20.0
Lithium	179	0.5	9.4	71.1	55.0
Magnesium	650	1.7	4.0	167	26.0
Molybdenum	2595	10.2	5.6	142	5.2
Nickel	1254	8.9	6.8	88.0	13.4
Palladium	1550	12.0	10.9	71.0	10.9
Platinum	1770	21.5	10.7	71.0	9.1
Rhodium	1959	12.4	4.7	84.0	8.4
Silicon	1414	2.3	$>10^{10}$	83.0	7.5
Silver	961	10.5	1.6	418	19.0
Tantalum	2985	17.0	14.0	54.0	6.5
Tin	232	7.3	12.9	65.0	12.0
Titanium	1670	4.5	55.0	17.0	8.9
Tungsten	3380	19.4	6.0	165	4.5
Uranium(a)	1130	19.0	29.0	29.3	23.0
Zinc	420	7.1	26.0	111	30.0
Zirconium	1860	6.5	45.0	20.0	5.9

Aluminum alloys are easily cut and formed by most mechanical methods. The softer aluminum alloys are very ductile and may be extruded, impact formed, rolled, or mechanically upset. Other alloys may be punched and formed to achieve a defined shape and then heat treated to achieve structural properties. Aluminum has a good strength-to-weight ratio and is used to achieve lightweight products. Commonly used aluminum alloy series include:

- 1100 Resists corrosion, good weldability, good conductivity, commonly heat treated, used for sheet metal parts, rivets, etc. without high-strength requirements
- 3003 Similar to 1100 except higher strength, more corrosion resistant, used for heat exchanger fins and like applications
- 5052 Excellent corrosion resistance especially in marine environments, higher strength than 3003, used for chassis parts, fan blades, and the like

- 6061 A heat-treatable high-strength material used for structural parts
- 356 series alloy Used for cast aluminum parts; may be heat treated and used for sand, permanent mold, and investment casting processes

Magnesium

Magnesium has similarities to aluminum. It is a good but slightly poorer electrical and thermal conductor with a high strength-to-weight ratio and is sometime used in place of aluminum to effect weight reduction. Care must be taken when using magnesium because magnesium corrodes readily, is highly reactive to oxidizing chemicals, and magnesium in shaving and powder form will oxidize (burn).

Copper and Its Alloys

The primary attributes of copper are its low electrical resistance and low resistance to the flow of heat (high thermal conductivity). Copper is used for conductive traces on printed circuit boards, in wiring, and in switches and relays. Copper may be machined, formed, and cast. It is easily soldered by conventional lead–tin electronic solders. Copper will tarnish and usually requires surface treatment to avoid tarnishing in service.

Copper is heavy, soft, and ductile thus not a good structural material unless used in an alloy with other materials such as beryllium. Beryllium–copper alloys are used for electrical contacts (improved wear) and are good materials for constructing springs and spring-type electrical contacts.

Cadmium, Chromium, Nickel, and Zinc

These metals are often used as an electroplated finish on iron alloy parts, such as fasteners, to improve the appearance of the parts and to achieve protection against galvanic corrosion.

Gold and Silver

Gold and silver are precious metals that exhibit very low electrical resistance and very high thermal conductivity. Both are used to electroplate connector, relay, and switch contacts to achieve low contact resistance, durability, and resistance to corrosion. Gold is an inert metal and not given to forming nonconductive surface films over time and thus is a good candidate for ‘‘dry circuit’’ switching. (A dry circuit is one where very low currents are involved, such as signals in a CMOS circuit. The low currents are insufficient to penetrate and dispel the effect of environmental-induced nonconductive films on the contacts. The result is a detrimental increase in contact resistance over time.)

Care must be used when silver is employed in a design because silver will easily tarnish; and, when used as switch or other contacts, this tarnish will create a nonconductive surface film on the silver part. Sulfur, such as found in brown wrapping paper, will quickly cause silver to tarnish. The application of antioxidant grease is used to protect electrical contacts fabricated from silver.

Both gold and silver are easily soldered using conventional electronic solders. When soldering to contacts that are silver plated, the solder will leach the plated silver from the wetted area resulting in degraded electrical performance. As a result, a "silver solder," containing approximately 2% silver, is be used to prevent leaching.

Lead and Tin

Lead is very heavy and may be used for shielding of electronic components that are subjected to ionizing radiation.

Tin is a soft and ductile material, substantially inert to the environment that has lubrication properties. Thus tin may be used to electroplate electrical contacts in connectors to improve the mechanical mating of connector elements.

Used together, lead and tin are the primary constituents in the majority of electronic solder alloys commonly used in the electronics industry. Lead–tin solders vary in the ratio of lead to tin in the alloy, with 63% lead and 37% tin being a eutectic mixture featuring the lowest melting point of solder and when cooling the entire mixture "freezes" at the same temperature, which leads to improved joint integrity particularly regarding the attachment of parts to a printed circuit board. Another common solder mixture consists of 60% lead and 40% tin. This mixture melts at a slightly higher temperature; however, it exhibits higher mechanical strength in the solidified form and, thus is used to attach wires to connectors and in other general applications.

The presence of lead in solder is believed to cause health risks, and lead-free solders[10] and lead-free conductive adhesives are being used in sensitive applications where lead must be avoided.

Rhodium

This metal has very good wear resistance and is used to coat electrical contacts subjected to repetitive usage, such as high-performance relay contacts.

Titanium

Heavier than aluminum but lighter than steel, titanium has a melting point comparable to steel, offers a relatively inert structural material with a high strength-to-weight ratio and is difficult to weld. Its electrical resistance is about 20 times that of aluminum, and its thermal conductivity is one-fourth that of aluminum. Its coefficient of thermal expansion is nearly the same as that of steel. Titanium is used for weight reduction when high strength is required for equipment attachments, mounting structures, and cases.

6.3 Plastics and Elastomers

Plastics or organic polymers find extensive use in electronic applications.[11] There are two basic types of plastics; thermoplastics and thermosetting plastics. The difference between thermoplastics and thermosets is whether or not the plastic is completely polymerized at the time of manufacture (i.e., a thermoplastic), or whether the plastic is provided as a resin plus a hardener, which must be mixed and polymerized when the plastic is formed into a component (i.e., a thermosetting plastic).

Plastics are commonly used in applications where electrical insulation is required or acceptable. Plastics are unable to operate over the temperature range of metals and are subject to softening at warm temperatures and embrittlement at cold temperatures. The physical properties vary widely, however, plastics are used in the construction of many electronic components and may be used for equipment housings and appearance hardware. Care must be taken when soldering near plastic parts, as touching plastic by a soldering iron tip can often cause local melting and deformation. Resistance to chemicals varies widely as does moisture absorption and mechanical load-bearing capacity. Most plastics are subject to creep and must be employed where permanent deformation due to sustained loading is controlled or may be tolerated.

Plastic materials may be formed into component parts by a wide variety of methods, and the cost per unit for large-volume production can be quite low. Typical plastics used in electronic packaging applications are identified in Table 4.

Thermoplastics

Thermoplastic materials are available in sheet, granules, and powder form; and may be fabricated by transfer molding, injection molding, laminating, pultrusion, compression molding, filament winding and pouring or casting. Thermoplastic materials can be softened by heat and merged or shaped. The properties of thermoplastics may be modified by the addition of fillers or reinforcing fibers.

ABS thermoplastics are tough and used to make enclosures, insulators, appearance sheet materials, and a variety of molded parts for electronic applications. They may be varied in color; however, the temperature range is limited. Most applications of the ABS thermoplastics are in consumer goods and other low-cost applications.

Acrylics may be used for conferral coating, and in solid form they are optically clear with very good weather resistance. They are temperature sensitive and will readily burn when exposed to flame.

Fluoropolymers are widely used in electronic applications due to the wide range of properties for which they may be formulated. They are highly heat resistant, are inert to most chemicals, have essentially zero water absorption, have a low coefficient of friction, have high arc resistance, resist creep, and have low dielectric losses. Teflon, as manufactured by DuPont, is an example of a fluoropolymer used for wire insulation, electrical insulation, low load bearings,

Table 4 Plastics Used in Electronic Packaging

Thermoplastics	Thermosets	Elastomers
ABS	Allyl	Natural rubber
Acrylic	Epoxy	Isoprene
Fluoropolymer	Melamine	Chloroprene, neoprene
Nylon	Phenolic	Ethylene–propylene
Polycarbonate	Polyimide	Fluorinated copolymers
Polyethermide	Polyurethane	Butyl
Polyethylene	Silicone	Nitrile, Buna N
Polyimide		PVC
Polystyrene		Silicone copolymers
PVC		Polysulfide
		Polyurethane

and dielectric purposes. Fluoropolymers are expensive, relatively soft, and hard to process.

Nylon is a polyamide polymer that is tough and abrasion resistant with good mechanical strength. It is used in fiber form to make fabrics and in solid form to make gears, standoffs, low-pressure unlubricated bearings, wire ties, connectors, wire jackets, and like items. Nylon absorbs water and its dielectric strength and resistivity varies with the humidity of the environment to which it is exposed. Nylon will degrade when subjected to sunlight and will exhibit creep or cold flow under sustained loads.

Polycarbonate materials exhibit high impact strength, are optically clear, flame resistant, and will perform in higher temperature applications. This material is used to make windows and other functional and appearance-related components. These materials absorb water and when under mechanical stress are degraded by many chemicals and solvents.

Polyetherimide polymer finds usage as fuse blocks, circuit board insulators, chip carriers, equipment housings, switch and circuit breaker housings, and like applications. It exhibits low dielectric losses, functions over a wide temperature range, has high radiation resistance, and withstands boiling water.

Polyethylene is an inert, abrasion-resistant olefin, with low weight per unit volume and has very low water absorption. This material is used in molded form for discrete component enclosures, battery cases, and like applications. In sheet or film form it is used as a dielectric in the manufacture of capacitors. It also is used as a vapor barrier and as insulation and sheath for wires and cables. When exposed to flame, olefins will burn.

Polymide plastic materials are used for radomes, antenna housings, films, printer circuit boards, and connectors. They have high radiation resistance, low coefficient of thermal expansion, high electrical resistance, are wear resistant, have a low dielectric loss factor and resist high temperatures. They are chemically resistant but are degraded by hot caustic solutions and highly polar solvents. Hard to mold, polymide shapes are formed by powder metallurgy and compression-molding techniques.

Polystyrene has a very low dissipation factor of about 0.0001 and about a 2.4 dielectric constant; thus, it is used in foam insulation materials and in radio frequency (rf) striplines and similar applications. It is degraded by many solvents and chemicals and by modestly elevated temperatures.

PVC, or polyvinyl chloride, materials are used to build wire sleeves and jackets, tubing, and insulation due to flexibility and resistance to most chemicals. They are sensitive to ultraviolet (sunlight) radiation, which causes them to become brittle and shrink. PVC use is restricted to temperatures below about 100°C, and it stiffens at temperatures below 0°C.

Thermosets

Thermoset plastic materials are supplied in the form of a resin and hardener, and the final polymerization of the plastic occurs during the final forming process. This final polymerization may be referred to as curing, hardening, vulcanizing, or cross-linking. Sometimes heat is required to complete or hasten the polymerization. The fabricator must be aware that the chemical process of curing involves the generation of heat by exothermic reaction within the material, and this heat must be shunted away to prevent cracking and other deterioration. The material also shrinks up to 10% by volume during cure, thus having an impact on the configuration of the mold. The addition of fillers, pigments, and lubricants may also be used to control shrinkage.

The allyl polymer family is characterized by good dimensional stability, having electrical insulation properties, the ability to withstand elevated temperatures, and good moisture resistance. A commonly used allyl includes glass-filled diallyl phthalate, which is dimensionally stable and is used to make inserts in connectors, electronic parts, housings, and to form insulating standoff posts.

Epoxy materials adhere well and are used as adhesives as well as for potting, casting, and encapsulation purposes. The properties may be varied from flexible to rigid. The final weight, strength, thermal conduction, and thermal expansion may be modified by inclusion of filler materials. Epoxies are used to encapsulate circuits such as microcircuit chips in multichip modules (MCM), chips-on-board (COB), and conventional components in cordwood array packaging. Epoxy resins are used with glass fabrics and phenolic laminates to fabricate printed circuit boards; they also are used for conformal coating, encapsulation, adhesives, and in forming durable paints and finishes.

Phenolic materials are used for high-temperature, high mechanical strength (relative to other plastics), and low-cost applications. Phenolics are used to fabricate knobs, handles, connectors, chip carriers, and in laminate form are used to build small enclosures. Phenolics are not elastic and may fracture upon bending or impact loading.

Polyimide materials exhibit low outgassing, good radiation resistance, and have very good high- and low-temperature performance. They must be used with care because they are sensitive to moisture and are degraded by organic acids and alkalis. Applications include bearings, seals, insulators, flexible cable, tapes, sleeving, moldings, wire enamel, chip carriers, and laminates.

Polyurethane materials are often used as paints, wire enamels, conformal coatings, embedding compounds and foam dielectric and thermal insulation. Some polyurethane materials may be poured and used as encapsulants to protect circuits from the environment. They exhibit elastomeric properties and have tear resistance, abrasion resistance, and unusual toughness. They can be degraded by strong acids and bases. The operating temperature range is limited and should not exceed 120°C.

Silicone-based materials have a wide range of general-purpose applications in electronic equipment. They may be formulated as sealants, elastomers, adhesives, paints, conformal coatings, and encapsulants. They have a broad temperature range (−40 to +250°C), have excellent arc resistance, are nonburning, and are water resistant when fully cured.

A commonly used silicone family is known as the room temperature vulcanizing (RTV) adhesives and sealants, which are often used to waterproof a component or electrical joint. These products polymerize with water when curing and in the process release acetic acid in some formulations or ethyl alcohol in other formulations. In applications where the presence of acetic acid vapor would degrade circuit elements such as switch and relay contacts, the RTV should be allowed to fully cure before sealing RTV within an electronic enclosure. Alternatively, if the presence of acetic acid on a component is a problem and ethyl alcohol may be tolerated, use of an RTV that does not exude acetic acid may be used in the design.

Elastomers

Elastomers[4] are substances, such as natural rubber and polymers, that have material properties that resemble rubber.[2,12] These substances have the ability to return rapidly nearly to their original shape after sustaining substantial deformation due to the application of mechanical stress and the release of that stress. The properties of elastomers are employed as sealing gaskets and drive belts and find other uses that involve bending and recovery of shape.

Sometimes used for gaskets and powertrain or drive belts, elastomers are much more vulnerable to aging and environmental degradation than other plastic materials and must be selected with care. The designer is cautioned to consider material properties such as compression set, creep, loss of elasticity, fatigue failure due to repetitive load, loss of elasticity at low temperatures, strength at elevated temperatures, tear resistance, and tensile strength that may be degraded by exposure to ultraviolet and ionizing radiation, elevated temperatures, exposure to some lubricants and chemicals, and continuous mechanical stress.

6.4 Ceramics and Glasses

Ceramics and glasses are used in similar ways in electronic packaging applications.[2] They have unique chemical, thermal, and mechanical properties. Both are primarily used as electrical insulators; however, glasses find a wider range of use to include substrates, capacitor and resistor bonding components, equipment and component enclosure seals, and similar uses.

Ceramics

Electronic ceramic materials include electrical porcelains, which have varied properties based on the amount of aluminum oxide incorporated into the material. Aluminum oxide is the additive of choice for ceramic materials for it improves mechanical strength, greatly improves thermal conductivity, and improves flexure strength. A concern, however, is that the use of aluminum oxide substantially increases the material coefficient of thermal expansion (about double that of silicon) and elevation of the material dielectric constant. Ceramics are often bonded to metal substrates to perform useful electronic functions.

Other oxides may be used such as beryllium (which is toxic) to achieve high thermal conductivity, aluminum nitride (which also has a high thermal conductivity) with thermal expansion nearly that of silicon, and boron nitride (which combines high thermal conductivity with machinability) in a softer yet durable material.

Glass

Glass is an amorphous noncrystalline material that may be heated, even to a liquid phase, and formed into useful shapes. Glass performs like a liquid that has been sufficiently cooled to become substantially stiff and rigid. The inclusions of additives can substantially alter glass melting point, thermal expansion, and electrical properties. Glass may be optically clear, fused with other materials to make resistors and capacitor elements, and is vulnerable to impact mechanical loads and thermal shock loads that cause glass to exhibit brittle fracture.

Soda-lime glasses are used to seal hermetic packages and to form insulator bushing for feed-through devices.

Borosilicate glasses have excellent resistance to chemicals, high electrical resistance and low dielectric constants, and are used as binders for compounds associated with component construction.

Lead alkali borosilicate glasses have a lower melting point and are used for adhesive and sealing applications, including semiconductor processing.

Glass ceramics are machinable materials that may have strength twice that of ordinary silicon dioxide glasses. They have a crystalline structure with temperature stability lower than that of other glasses and may be shaped into a variety of useful configurations.

6.5 Adhesives

Adhesives are materials that by adhesion, cohesion, or molecular bonding cling to the surface of another material. Adhesive materials used for electronic applications are most often derived from elastomers and plastic polymers. Glasses are used to perform adhesive functions when the properties of elastomers and plastics have inadequate temperature range or are insufficiently inert to the product environment. Lead–tin solders are used as conductive adhesives in the application of surface-mounted parts to printed circuit boards.

Adhesives are utilized for structural bonding, to seal enclosures, to bond and protect components from the environment, to seal mechanical joints and thus prevent galvanic corrosion, to join parts for less cost than with mechanical means, and to join materials where the temperatures developed by processes such as brazing or soldering would degrade the bonded elements.

The success of an adhesive application is at least as dependent upon proper joint design and application procedures as it is on the selection of a specific adhesive material.

The primary requirements for achieving a successful adhesive joint include:

- The area of the adhesive overlap must be adequate to withstand the mechanical loads imposed in service.
- The mechanical loading generally should not provide peel or cleavage stresses on the adhesive joint.
- The adhesive must be able to wet and bond to the substrate material to which it is applied.
- The surfaces to be joined must be chemically clean and may require a properly applied primer.
- If the adhesive mixture consists of multiple components, these components must be accurately measured and properly mixed.
- Pressure, elevated temperatures, and positioning fixtures may be needed during cure of the adhesive.

Table 5 Adhesives Used in Electronic Packaging

Thermosetting	Thermoplastic	Elastomeric	Hybrid
Cyanoacrylate	Polyvinyl acetate	Butyl	Epoxy phenolic
Polyester	Polyvinyl acetal	Styrene butadiene	Epoxy polysulfide
Epoxy	Polyamide	Phenolic	Neoprene
Phenolic	Acrylic	Polysulfide	Vinyl phenolic
Polyimide		Silicone	
		Neoprene	

- The cure cycle may require several hours for the adhesive to gain full strength.
- The adhesive material must withstand processing and usage environmental temperatures and chemicals.

There are three categories of adhesive materials:

1. *Structural Adhesives* intended to hold two or more parts together under conditions where the adhesive joint is subjected to high mechanical loads
2. *Holding Adhesives* intended to permanently or on a temporary basis hold lightweight items in place
3. *Sealing Adhesives* intended to fill a space between two or more materials and provide a seal without the need to have high structural strength

There are a wide variety of materials utilized as adhesives that include at least the adhesives identified by Table 5.

7 SUMMARY

The key to successful materials selection is the ability to identify the set of materials most likely to offer a design solution and then to correctly select and employ one or more materials from that set to provide the optimum result. This involves the careful definition of the dominant technical functions that the material must perform including compliance with an overriding set of considerations. A review of the use of materials in successful design applications is also a useful guide to materials selection. Within these constraints, the total cost of use [basic materials cost, material preparation (machining, molding, forming, joining, finishing, assembly, packaging, handling, protection, service limitations, other) and customer support issues] may be determined or estimated. Each of the remaining materials selection options may then be compared, thus leading to identification of the most economical and suitable choice of material for the application.

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CHAPTER 40

ADVANCED MATERIALS IN SPORTS EQUIPMENT

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1 INTRODUCTION

Advanced materials can significantly enhance sports performance and dramatically tilt the playing field. This paper discusses the use of advanced materials in sports and suggests that there are ethical questions surrounding their use.

Advanced materials with mechanical and physical behavior characteristics well in excess of those exhibited by conventional high-volume materials such as steels and aluminum alloys have contributed significantly to the increased performance of transportation systems in aerospace, automobiles, and rolling stock (trains). The important characteristics include strength, ductility, stiffness (modulus), temperature capability, forgiveness (a collective term including fracture toughness, fatigue crack growth rate, etc.), and low density. For many high-performance applications high cost can be accepted, although the level of acceptance depends upon the industry in question (Fig. 1).

In this chapter the role of advanced materials in a number of sporting events will be addressed. At the highest professional level sports are a highly competitive occupation with millions of dollars depending upon fractions of a second or tenths of an inch. Even the dedicated amateur is willing to invest a great deal of money to improve performance, even though this may occur as infrequently

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Handbook of Materials Selection. Edited by Myer Kutz

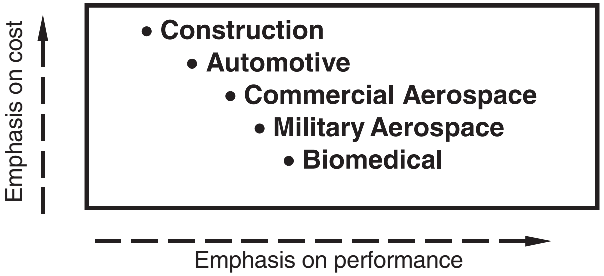

Fig. 1 Impact of cost in various industries.

as once a month. Thus, just as in the industries mentioned earlier, if advanced materials lead to enhanced performance, their use can be justified; and a $17 billion sporting equipment market in the United States in 1997 says the buying public agrees (Fig. 2). The characteristics of advanced materials that lend themselves to this enhanced sporting behavior parallel those listed for other industries above.

Just as in the transportation industry the materials of choice for sports have shown a major evolution over the last 100 years. From naturally occurring materials such as wood, twine, gut, and rubber we have progressed to high-

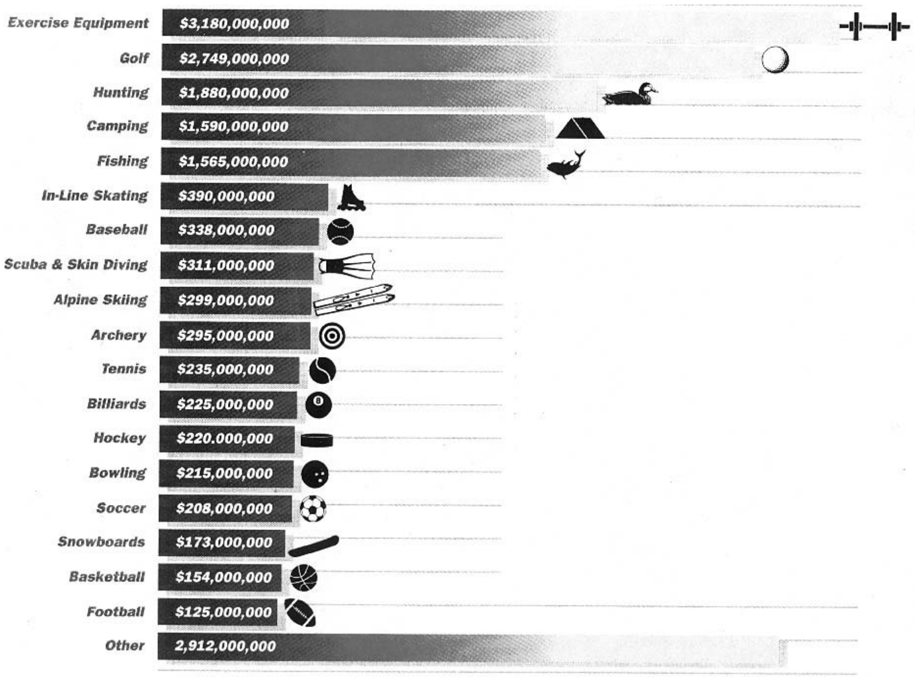

Fig. 2 Graph showing how industries in advanced materials lead to enhanced performance, and a $17-billion sporting equipment market in the United States in 1997 says the buying public agrees.

technology metals, polymers, ceramics, and synthetic hybrid materials, including composites and cellular concepts.

In this chapter consideration is first given to a broad discussion of the mechanical properties of materials that are significant in sporting equipment. Then there is a more detailed examination of how advanced materials have impacted various specific sports. Here an attempt has been made to define how measurable (absolute) records have been influenced by these advanced materials. I have attempted to subtract the portion of these improvements that are a result of improvements in the capability of the human body, whether this be a consequence of enhanced body function derived from superior training, diet, or will power. Advanced materials have led to substantial improvement in some sports, much less in others. In addition, advanced materials have not only led to improvements in performance for the paraplegic athlete, but in some cases participation would not have been even possible without them. Finally, there is a discussion of the ethics of the use of advanced materials in the sporting arena.

The term *technological momentum*[1]—once technological choices are made (or ‘‘allowed’’) and implemented, reversing the decision becomes difficult—exists for sporting equipment. This is particularly the case when athletes and manufacturers have a great deal invested in the new technology. Once a sports organization makes a technological choice, it is often a permanent decision.

2 CHARACTERISTICS OF MATERIALS OF IMPORTANCE IN SPORTS EQUIPMENT DESIGN

The optimum design of sports equipment requires the application of a number of disciplines not only for the enhanced performance already mentioned but also to make the equipment as ‘‘user friendly’’ as possible from the standpoint of the avoidance of injuries.[2] Clearly sports equipment design encompasses materials science, mechanical engineering, and physics; however, it also necessitates a knowledge of anatomy, physiology, and biomechanics. Biomechanics can be simply defined as the science of how the body reacts to internal and external forces.[3] It is thus an attempt to apply the basic laws of physics and mechanics to the joints, ligaments, and tissues of the body as they are subjected to loading (Fig. 3).

In designing sports equipment, various characteristics of materials must be considered[4,5]:

- Strength
- Density
- Ductility
- Fatigue resistance
- Toughness
- Modulus (damping)
- Cost

To meet the requirements of sports equipment, the materials of choice often consist of a mixture of material types, metals, ceramics, polymers, and composite concepts. They are fabricated into the desired equipment making use of creative

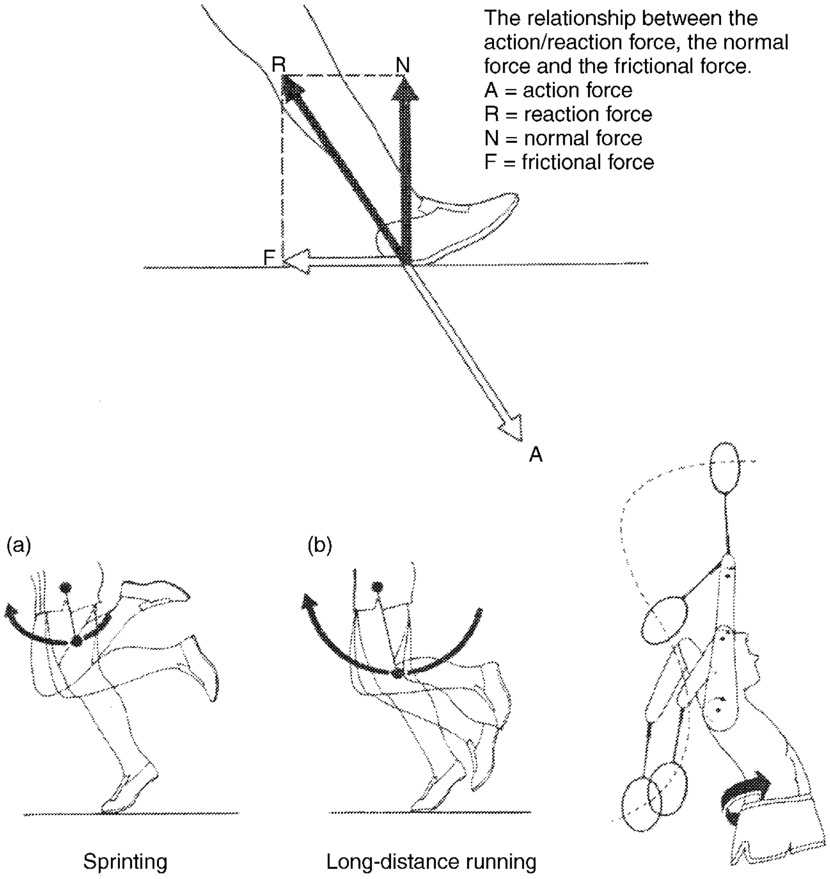

Fig. 3 Biomechanics of sports addresses the analysis of forces and stresses which act on the body in various sports. (From Ref. 2.)

design concepts with due attention being given to biomechanical requirements. By comparing specific properties (i.e., taking the difference in density of competing materials into account), the attributes of different materials can be better evaluated (Table 1). If we want a material that features the highest possible stiffness for the least possible weight, we would select the materials with the

Table 1 Typical Mechanical Properties of Material Classes

Materials Classes	Young's Modulus E (MPa) × 10^3	Density ρ (mg m^{-3})	$E/\rho \times 10^3$
Metals	40–210	2–8	24–30
Glass	73	~2.5	~30
Ceramics	400–700	~3.5	100–230
Fibers (B,C)	~400	~2.4	~170
Carbon fiber composite	200	2.0	100
Wood	14	0.5	28

highest specific stiffness. Cellular concepts win out compared to monolithic materials in this regard because the density of cellular materials are less than those of solid articles. In the actual design of complex sports equipment, a specific design criterion needs to be defined to allow the optimum materials selection to be made.

3 THE IMPACT OF ADVANCED MATERIALS ON SPORTS PERFORMANCE

To illustrate how advanced materials have impacted sports performance a number of sporting events will be considered in which a contribution to the improved performance can be attributed to the materials used to construct equipment. Wherever possible, measurable quantities (distance, time) of improvements in performance will be given.[6]

3.1 Running

Shoes have provided substantial improvements in the running events. The human feet are very complex biomechanical structures that are highly prone to stress and injury.[2] Thus, a running shoe needs to be complex and consists of a variety of different materials (Fig. 4),[6] which are selected according to their resilience, strength, elasticity (stretchability), compression, durability, and wear resistance. About 80% of runners hit the ground on the center heel, roll onto the midfoot, and finally push off with the ball of the foot.[2] The midsole is key in providing cushioning during impact with the ground. Usually, it is made from a plastic foam that in some concepts includes "air pockets" (Air Max) filled with pressurized gas. However, these cushioning effects break down with use (even after only 100 km of running) with reduced cushioning efficiency. For the future, better cushioning systems are likely with increased tailoring to the individual (if you can afford it).

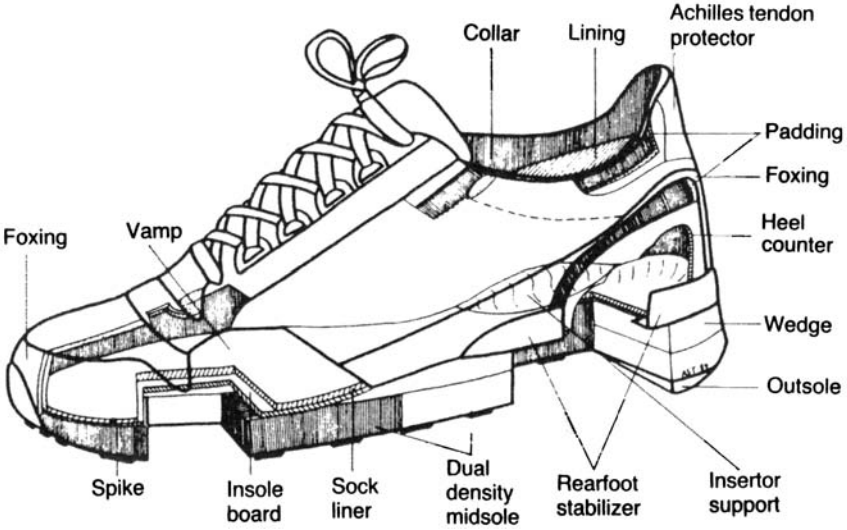

Fig. 4 Parts of a running shoe. (From Ref. 2.)

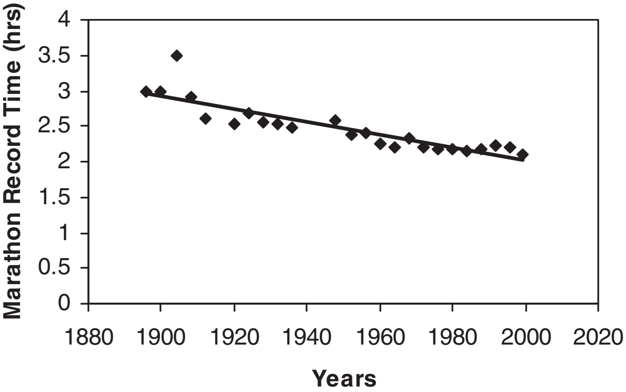

Fig. 5 Improvement in Olympic records for the men's marathon in the past 100 years.

This author contends that the improvements summarized above have been much more in the comfort/avoidance of injury arena than in absolute performance enhancement. In 1896 in the first modern-day Olympics when Spiridon Loues won the marathon (which was actually somewhat shorter in distance than it is today), for all of Greece to celebrate with him, he barely broke 3 h (Fig. 5). Almost 100 years later the Olympic marathon record is a little over 2 h, about a 30% improvement. The majority of this improvement can be attributed to an improvement in human performance. The same can be said for the 100-m event (Fig. 6).

If we turn our attention to the paralympics, a totally different situation exists. There were no Olympic Games in 1896 for those requiring prosthetic limbs. The paraplegic could hardly move, never mind compete in athletic competition. But by 1992 the paraplegic could not only compete, but he could also outperform the majority of us who have use of all our limbs.

Paraplegic Joe Gaetani broke world records in both the 100-m and 200-m sprint events in Barcelona (Table 2). He made use of the amazing Springlite II

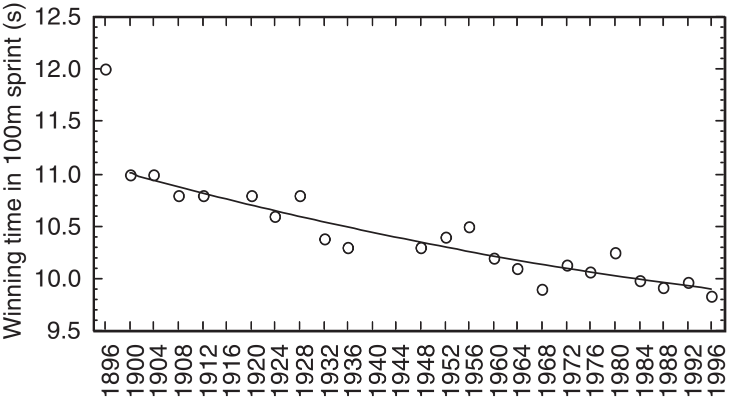

Fig. 6 Winning times for the 100-m sprint at the Olympic Games since 1896.

Table 2 World Records Set by Joe Gaetani in the 1992 Paralympics in Barcelona[a]

Event (m)	Time (s)
100	12.23
200	24.82

[a] Bilateral below-knee amputee athlete (Class A-3).

prosthetic device, which features a thin carbon fiber/epoxy pylon that provides the right balance of stiffness and flex at a substantially reduced weight compared to conventional materials (3 lb compared to 10 lb) such as wood. An energy storage return aids performance and contributes to the capability to walk or run greater distances with much less fatigue and discomfort than with traditional devices. The cost of the Springlite II is about $850.

Born without feet, Tony Volpentest won gold medals in Barcelona in the 100-m (11.63 s) and the 200-m (23.07 s) events. He runs on carbon–graphite feet bolted to carbon composite sockets that encase his legs (built by Flex-foot Inc.), the arrangement acts like a spring-board. With each step, the runner punches the track, which catapults him forward more efficiently than if he were running on two human feet. And the long-distance runner is not forgotten: This device is stiff and springey for sprinters but shock-absorbing for the marathoner.

3.2 Pole Vaulting

The requirements for a vault are shown in Table 3. The 1896 Olympics saw a height of 3.30 m achieved with a bamboo pole in the pole-vault event. Figure 7 shows the winning heights in the pole-vault discipline for all Olympic events since the first. The bamboo pole, which has more spring and is much lighter for the same stiffness than the hickory pole, was introduced in 1904 by an Olympiad from my hometown of Moscow, Idaho, A. J. Gilbert (Dan O'Brian is the second Olympian from our town of 18,000). Initially this change in materials provided an advantage of about 200 mm in height. Improvements in coaching and technique allowed a gradual increase in height over the years. By the late 1950s, however, the gains were starting to level off and lighter weight aluminum poles were used for a short time. The greatest performance improvement occurred in 1964 with a substantial gain of about 250 mm over the previous Olympics, to a height of 5.10 m. This improvement was caused by the introduction of glass-fiber composites, which were lightweight and had higher stiffness. Not only was this new material more efficient, it allowed athletes to change their style by

Table 3 Requirement for a Vaulting Pole

Requirements	Possible Materials of Choice
Light (low density)	Bamboo
Buckling resistance (stiffness)	Aluminum
Strong (resistance to plastic strain)	Steel
Minimal twisting (high torsional strain resistance)	Magnesium
Cost	Carbon fiber composite

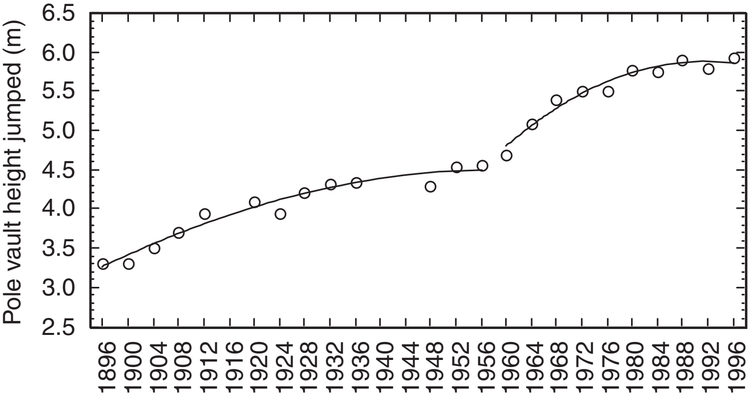

Fig. 7 Winning heights in pole vaults since first Olympic event.

turning upside-down and gliding over the bar feet first. Improvements in technique using the new pole once again lead to increases in performance that are only just beginning to slow down.

If we refer to Table 1, and also add the requirement of durability, the material of choice is the carbon fiber composite, with bamboo not too far behind. If we add in the minimal twisting requirement, the carbon fiber composite becomes even more attractive (Fig. 8) with three layers of different fibers being used to optimize the performance. An outer layer of high-strength carbon fiber provides high stiffness, while an intermediate webbing of fibers together with an inner layer of wound glass fiber, builds in resistance to twisting. The glass fiber consists of 80% longitudinal and 20% radial fibers. So for the pole vault advanced materials have a major influence on performance.

3.3 Bicycling

Cycling is a highly efficient form of transportation, with the energy consumption lower than for walkers, and much lower than for powered vehicles.[2,7,8] The Chinese, recognizing this, build over 10 million bicycles per year. The bicycle

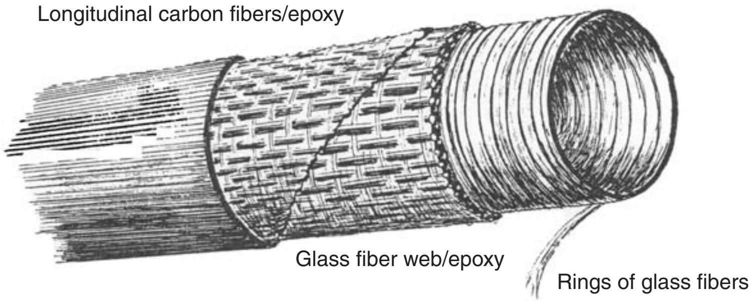

Fig. 8 Adding in minimal twisting requirement, carbon fiber composite becomes more attractive with three layers of different fibers being used to optimize the performance. (From Ref. 2.)

has been around for almost 200 years maturing from the 1817 Draisienne (walking device) to the modern-day Rover Safety Bicycle designed by J. K. Starley in 1885. Nowadays, advanced materials, in combination with aerodynamic considerations, have led to vastly improved bicycles. When Greg LeMond won the 1989 Tour de France (3420 km) by only 8 s, for example, his success was made possible by a clip-on extension of his handlebars with a padded tube, which enhanced his aerodynamic shape.

The bicycle can be considered to be a modified "space frame" such as that found in bridges, cranes, etc. For the bicycle this is the "diamond frame" (Fig. 9).

A number of advances have contributed to the high efficiency of a modern-day bicycle including the development of spoke wheels, the chain concept, pneumatic tires, and such accessories as seats, brake levers, and pedals. The two major advances are in the frame and wheels, however, and we will now consider these two components of the advanced bicycle.

Frame

The diamond frame and the alternate structure, the "cross frame," are both constructed from thin-walled tubular components that must resist tension, compression, bending, and torsional stresses. The requirements for the materials of construction of a bicycle are shown in Table 4.

These materials requirements can be simplified to the minimization of the bending of a cantilever beam; then the mass is given by

$$M = \text{const} \times \frac{\rho}{E}$$

Thus the optimum material is the material with the highest specific strength ρ/E. Based upon the data shown in Table 1, carbon fiber composites are the

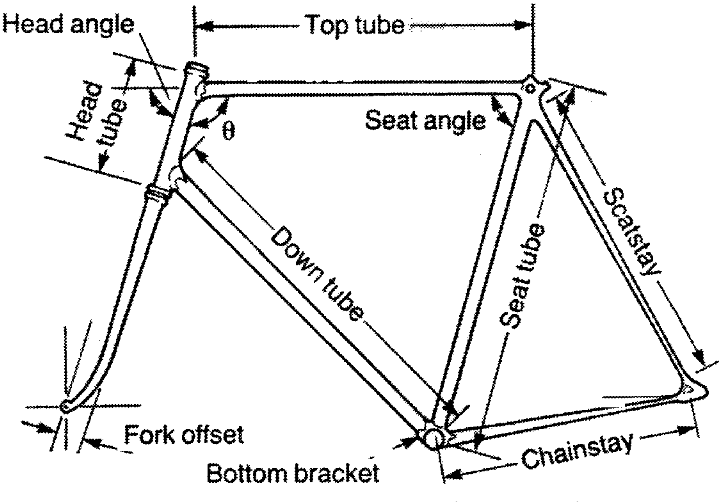

Fig. 9 Bicycle diamond frame. (From Ref. 2.)

Table 4 Materials Requirements for a Bicycle and Possible Materials of Choice

Requirements	Possible Materials of Choice
Light (low density)	Carbon fiber composites
Stiff	Aluminum
Strong	Titanium
Toughness	Magnesium
Fatigue resistance	Steel
Corrosion resistance	Metal matrix composites
Cost	

materials of choice if there is no concern over cost. Lightweight metals—aluminum, magnesium and titanium—are also attractive, as are metal matrix composites (Fig. 10[9]). If cost is a concern, then steel, which is not that far behind the other materials, is the obvious material for selection.

In addition to the carbon fiber reinforced composite frames, recently frames have been produced from magnesium, aluminum, titanium and metal matrix composites. In addition hybrid frames such as carbon fiber reinforced composite combined with titanium have been produced.

Wheels

Advances here include wheels with increased stability and rigidity for off-road bikes constructed from glass fiber reinforced nylon, and disc wheels. In the latter concept discs made of aluminum alloys or carbon fiber reinforced composites replace the spokes in conventional wheels. Developments also include three or five spoke wheels for rigidity and crosswind aerodynamics.

The improvements that advanced materials have produced in bicycling can be gauged in the enhancement in the Olympic pursuit records shown in Table 5.

Fig. 10 Road bicycle made by Harry Havnoonian with a mix of materials using adhesive bonding for joints, investment-cast stainless steel lugs, Ti-3Al-2.5V head tube, SiC-fiber-reinforced aluminum alloy front triangle, CRFP rear triangle, and CFRP rear wheel.

Table 5 Improvement in Olympic Records for the Bicycle 4000-m Individual Pursuit

Competitor	Year	Time
Daler (CZE)	1964	5 min 5 s
Boardman (GB)	1992	3 min 22 s

3.4 Tennis and Squash

Tennis is a sport in which absolute achievements cannot be compared. Who can argue with the notion that Bobby Riggs with his 1939 wooden racket would have no chance against Pete Sampras armed with an oversized composite racket with an enlarged "sweet spot." In fact, this confrontation has been framed as "like fighting against rifles with bows and arrows."[10]

Until about 25 years ago, tennis rackets were made from wood with ash, maple, and okume leading the way. In the late 1960s metal frames, generally fabricated from steel or aluminum, were introduced. At the present time, composite rackets are all the rage, not only from the viewpoint of efficiently accelerating the ball across the net, but also in terms of damping dangerous vibration, which can lead to "tennis elbow" (Fig. 11).

The impact force experienced by a player on returning a tennis ball traveling at 160 km per hour (100 mph) is approximately equivalent to jerk-lifting a weight of about 75 kg (170 lb). These forces can transmit a high load to the lateral epicondyle, located on the outer side of the elbow; leading to damage to the small blood capillaries in the muscles and tendons around the elbow joint. Better technique can help, but improved rackets can also make a major contribution.

The goal in designing modern tennis rackets is to increase the size of the sweet spot, the central part of the racket, which leads to little or no shock to the player and minimal vibration occurs upon impact with the ball. This depends

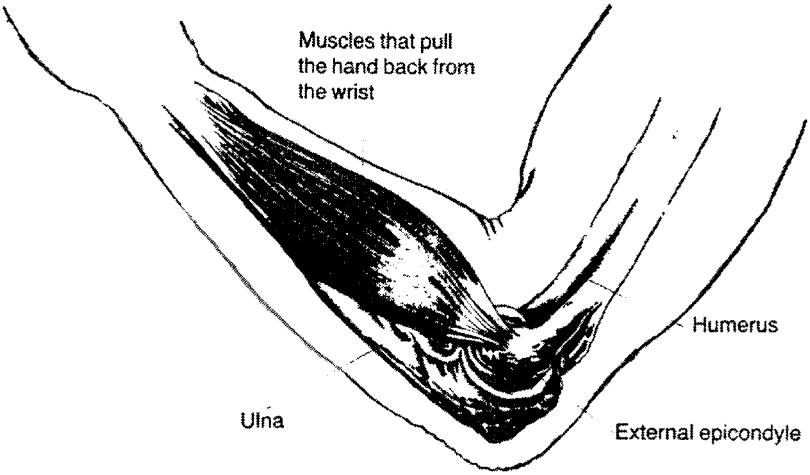

Fig. 11 Dangerous vibration can lead to tennis elbow. (From Ref. 2.)

upon the stiffness of the frame and the size and shape of the handle and head. The International Tennis Federation have now imposed an upper limit to the size of the racket.

Today tennis rackets are produced from monolithic metals, including steel, aluminum, magnesium, and titanium and metal matrix composites. However, the high stiffness of carbon-fiber-reinforced composites makes them superior to the metals in imparting high forces to the ball. To reduce the high-frequency vibration upon impact, racket handles are constructed of multiple fiber-reinforced layers wrapped around a soft inner core, which is often an injected polyurethane foam or honeycomb construction.

An example of a state-of-the-art tennis racket is the Wilson FPK, which consists of a urethane core, 84% graphite, 12% Kevlar, and 4% fiber FP (a pure form of ceramic aluminum oxide). The graphite provides strength and stiffness, thereby minimizing head deflection and helps to prevent twisting of the racket head when the ball impacts outside the sweet spot. The Kevlar fibers lead to additional strength and durability and contribute to damping vibration. The fiber FP produces even greater stiffness and damping to this type of tennis racket.

Squash rackets have shown similar trends to tennis rackets.[2] Until 1983, the frame was constructed of wood. Since then, squash rackets have featured hollow extruded aluminum designs and fiber-reinforced plastics, the latter featuring lightness, strength, toughness, and reduced vibration.

Racket strings have transitioned from ox gut to the modern-day synthetic (e.g., nylon) strings. This transition has not been without controversy. In the late 1970s, so-called spaghetti stringing, which resulted in large amounts of spin being imparted to the ball and an unpredictable bounces, led to a rule requiring ‘‘strings to be interlaced and attachments for durability purposes only, not designed to alter the flight in the ball.’’[11,12]

Wheelchairs (Fig. 12) now allow the paraplegic athlete to compete in this sport, as well as others, including racing events, basketball, and rugby. No heavy steel frames here—rather bikelike wheels, use of aerospace carbon fibers, and titanium, as well as computer-aided design of the suspension. Now there is a chair for each sport: basketball, racing, and even tennis. For example, tennis chairs are built with sharply slanted back wheels so the athlete can move quickly from side to side. In basketball, forwards have high seats, guards have more slant in their chairs in order to turn quickly. Cost? Top wheelchairs are in the range of $2000–$3300 apiece.

3.5 Cricket

As with tennis rackets, the manufacturers of cricket bats have been concerned with the size of the sweet spot and the reduction of flexural vibrations.[13] Three significant modes of flexural vibrations detract from the ideal rigid-body performance, and whereas distributing the weight of the blade to the edges (perimeter weighting) does not increase modal frequencies significantly, it may increase the width of the sweet spot.

3.6 Golf

Paralleling the tennis situation, it is very difficult to compare many of the achievements of the past with those of today in absolute terms. Clubs have

Fig. 12 Steve Meredith of Titanium Sports of Kennewick, WA (left) along with Alistair Godfrey and Malcolm Ward-Close of the Defense Evaluation and Research Agency (DERA), United Kingdom, are shown inspecting a titanium wheel chair.

evolved tremendously and it is difficult to imagine that Bobby Jones, using hickory shafts, could compete, at least in length, with Tiger Woods, Ernie Els, or David Duval armed with a shaft constructed from a graphite-epoxy and an over-sized hollow titanium head; albeit at a high price.

The materials evolution for the driver is dramatic. The overall weight has been decreased and the length of the club increased from 100 cm to as much as 125 cm. The grip and shaft weight has been reduced from 165 grams down to 115 grams or less. The weight of the head remains the same at about 200 g but using a hollow titanium (casting) construction, the head is now much bigger, with the mass concentrated around the outside of the hitting face. The net result is a club that is claimed to give greater distance (greater club head speed because of the longer arc) but also a straighter (bigger sweet spot) shot.

A concern of the U.S. Amateur Golf Association (USGA) is that technology may dominate over skill, i.e., no “spring-back” (trampoline) effect should occur when the ball is struck by the clubface, which may add distance. When a golf ball hits any surface it rebounds at a velocity less than that at which it hit the surface (Physics 101).[14] Materials with a high elasticity and high strength result in a high rebound velocity (or high coefficient of restitution [COR]), especially when the impacted surface is thin (allowing the “trampoline” effect). Thus the hitting surface can be tuned to increase the COR. However, this now puts us on the ragged edge of the mechanical property limits of materials of construction. Thus you will hear of the “proprietary heat treatments” (simply an aging treatment to increase the strength) to create a beta titanium alloy that is strong and capable of resisting fracture (most of the time) even though it is thinner than conventional titanium (e.g., Ti–6Al–4V) hitting faces.

With a tuned (nonconforming) face, the COR will exceed the USGA limit of 0.83 (somewhat arbitrarily set to allow the majority of the drivers at the time the limit was set to meet the requirement). However, the salient question is: Do the nonconforming drivers actually lead to increased distance? Tests by *Golf Digest*[14] indicated that the trampoline effect is maximized only in a relatively small area of the clubface. This means that it requires the impact accuracy of a robot or a professional to take full advantage of the effect. This has been confirmed in robot testing of a nonconforming club head (COR 0.845) where a 2% increase in ball speed (over a confirming club with a COR of 0.81) increased carry distance by 1–2 yards for 220 yards shots and 4–6 yards for 270-yard drives (the higher the swing speed, the greater the increase in ball speed and therefore, carry distance). And to the crux of the matter: 40 players (0 to 22 handicap) had essentially the same distance from both the conforming and nonconforming clubs. Worse: With off-center hits, there are indications that the nonconforming club hits the ball shorter distances than the confirming club.

The USGA fears that “trampoline” clubs and other “out there” technologies could add 30 yards to a drive.[15] With a perfect center hit, a COR of 0.880/0.900 (the maximum likely) and a 109-mph swing (a pro’s swing), the distance gap between a persimmon wood (COR 0.77) is about 25 yards.[15] So potentially a maximum of another 25 yards is possible with a nonconforming driver (COR 0.880/0.900) with an optimally struck approved golf ball. Thus there is a legitimate concern with the professionals, probably none for the average golfer.

Driving distance is an absolute criterion, which can be tracked versus the material of construction, with a caveat that the modern top golfers on the PGA tour now realize that exercise and body-building help their performance, far from a common practice in the past. The average PGA tour driving distance is shown in Fig. 13,[15] with a spike in 1994 at the time of the introduction of the titanium driver. A complication here is that balls have also evolved tremendously, with the most lively being banned because they would obsolete current golf courses.

3.7 Baseball/Softball

Aluminum baseball bats are banned in the U.S. major leagues because they would make current baseball stadiums obsolete. There would be too many home runs. However, both new aluminum bat concepts such as the ultralight, with a double-walled barrel construction, and titanium bats are revolutionizing softball. These bats have bigger sweet spots and lead to greater velocity off the bat. However, the Softball Association is concerned with an increase in injuries to infielders who cannot react quickly enough to this higher velocity. An example of a softball bat produced from titanium using a powder metallurgy approach is shown in Fig. 14.

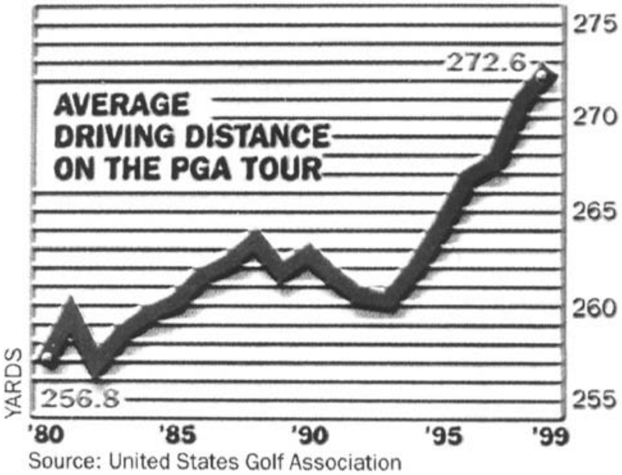

Fig. 13 Average driving distance on the PGA tour. Note spike since the introduction of the titanium driver in 1994.

3.8 Boats, Boards, and Wind-Surfing Fins

For all watercraft, there are four basic forces to contend with when considering design: weight, lift, thrust, and drag (Fig. 15).[2] Materials of construction are needed that result in lightweight, low skin friction (to give smooth flow and reduced drag), increased toughness and a high level of safety. Modern craft consist of a combination of several polymeric composite materials, often incorporating cellular (sandwich) concepts.

The construction of a wind-surfing board is complex consisting of a core of extruded foam polyestrene filler enclosed in fiberglass. This core is covered

Fig. 14 Susan Abkowitz of Dynamet Technology, Inc. holds a softball bat with a titanium alloy outer shell.

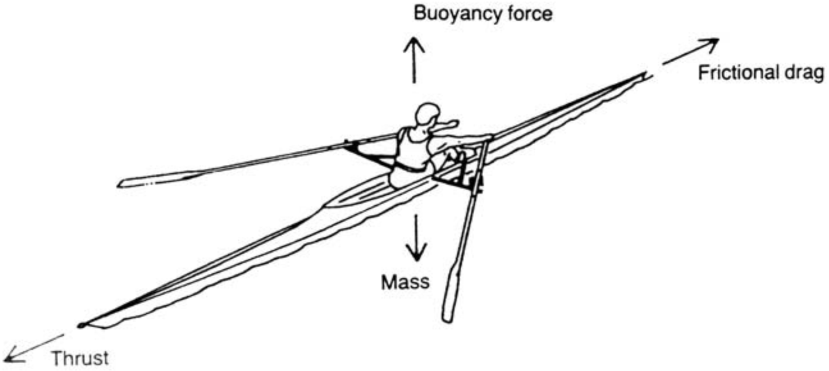

Fig. 15 Forces acting on a watercraft.

with graphite and glass fibers embedded in resin matrices, with an outer surface of glass-fiber-reinforced composite.

An interesting materials application involves the design of the wind-surfing fin. The challenge is to design a fin with optimal hydrodynamic shape because surfers need to perform equally in both directions, requiring a symmetric design. Innovation relied on a concept known as hydroelastic tailoring.[16] Through this process, structural deformation of the cross sections (Fig. 16) is induced by the hydrodynamic–pressure forces, with the internal structure encased in a flexible elastomer material covering. As this section moves through the water (at an incidence angle), a surface pressure loading results, as shown. This surface pressure loading is of insufficient magnitude to deform the supporting internal struc-

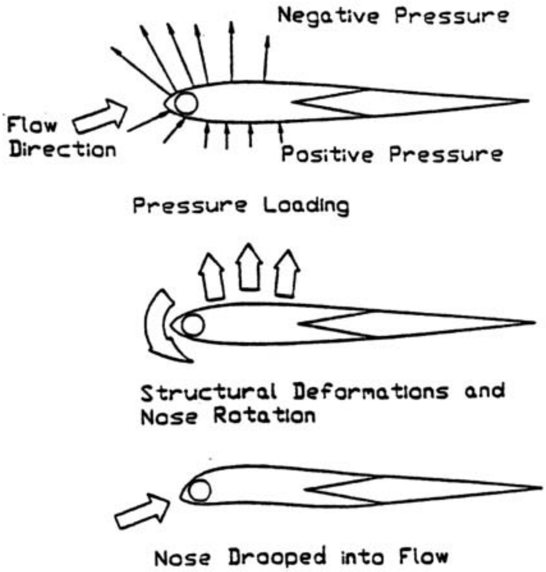

Fig. 16 Method of operation of hydroelastically tailored wind-surfing fin.

ture but is sufficiently strong to deform the elastomer region. As a result, the cross section will assume the cambered geometry shown. If a satisfactory shape is achieved, the end result will be an increase in lift-to-drag ratio of the fin—one of the design parameters used to improve the fin performance. The material of choice here is carbon fiber/Kevlar polymer composite.

3.9 Javelin

The javelin was an event enjoyed by the Mycenaeans at least 3000 years ago.[17] The Greeks of 500 BC used a thin wooden javelin with a cord wrapped around its center of mass. When thrown, the thrower held onto the end of the cord to make the javelin rotate through the air. The rotation acted to stabilize the javelin by averaging any non-symmetry in its construction about a central axis.

The modern javelin has relatively strict rules concerning its construction.[17] Essentially, the modern javelin must be smooth and has strict geometric rules to ensure the positioning of the center of mass. The reason for this can be seen in Fig. 17 which shows winning throws at the Olympic Games from 1904 onward. At the Athens Olympic Games in 1908 the winning throw was just over 50 m. In 1984, Uwe Hohn (from the then East Germany) threw a staggering 104.80 m. Given the dimensions of stadia and the fact that it was becoming unsafe for spectators, it was decided fairly quickly by the International Amatuer Athletics Federation that the javelin had to be redesigned to "under-perform." This was done by moving the center of mass forward by 4 cm, which caused a dramatic loss in lift and a consequent reduction in the distance traveled.

Figure 17 shows that the distances for the new-rules javelin were approximately 15 m less after the center of mass rule change. As far as the rule makers are concerned there are two advantages: (1) the javelin does not fly as far and (2) the javelin clearly lands tip first. Although this reduces overall throw distances, the new rule does give the athlete one advantage. The old-rules javelin was very sensitive to the initial throw conditions and even a small change could reduce distances by 20 m. The new-rules javelin is much less sensitive to initial conditions partly because it always has a negative pitching moment. This is likely to allow the athlete to produce more consistent throws.

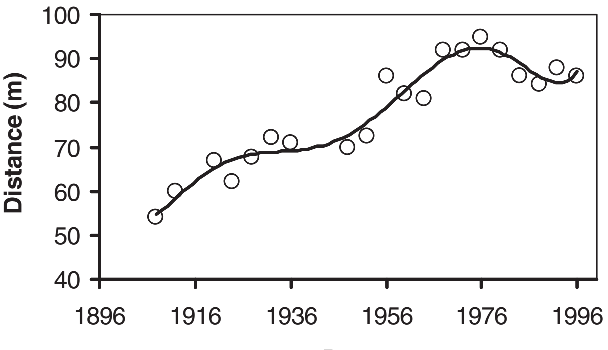

Fig. 17 Winning Olympic Games javelin throws.

As with the pole vault, technology strongly influences the performance of javelin throwers, and the IAAF successfully used an equipment change to reduce the length of throws from the mid 1980s. As Fig. 17 shows, however, it might not be too long until a further rule change is required!

3.10 Skiing and Boards

Skis need to provide for good longitudinal torsional rigidity to allow for good weight/pressure distribution while being flexible enough to respond to different surface properties (snow conditions) to dampen vibrations that could lead to hip and knee injuries (Fig. 18).[2] Modern skis consist of a cellular core (of polymeric or metal construction) and layered material around this core (including metals and polymer composites reinforced with Kevlar, aramid, and carbon fibers) with metal edges of steel or high-strength aluminum alloys. Debatably, the top-of-the-line skis, at the present time is the Volant titanium model, again with steel edges for high hardness, toughness, and wears resistance[18] (Fig. 19).

3.11 Hockey Equipment

The hockey stick has changed dramatically over the years, transitioning from wooden handles and paddles to hybrids of wood and fiberglass composites, and recently to include aluminum and carbon or graphite composite shafts.[19] The latter concept giving improved performance and player comfort.

4 ETHICAL CONSIDERATIONS

The use of advanced materials in sporting goods has been discussed with some examples being given; but certainly not all that are possible. Where the sporting achievement can be gauged in absolute terms, tremendous improvements have been made in those sports where equipment is critical. However, use of advanced materials in sports equipment presents us with some ethical questions.[20] We can clearly enhance behavior by allowing the use of advanced materials, but where

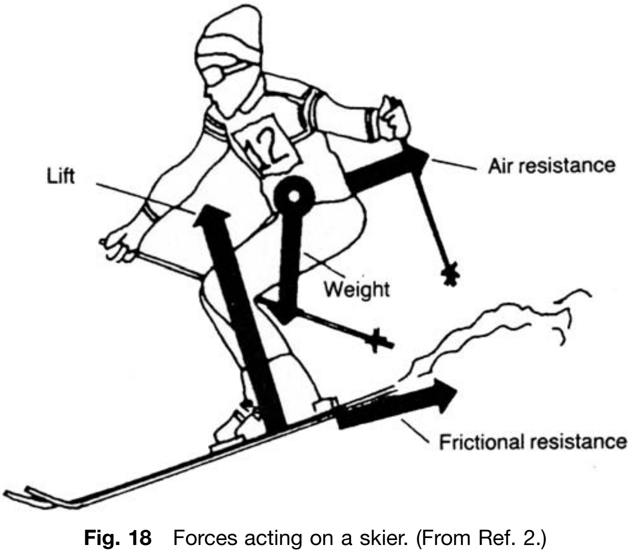

Fig. 18 Forces acting on a skier. (From Ref. 2.)

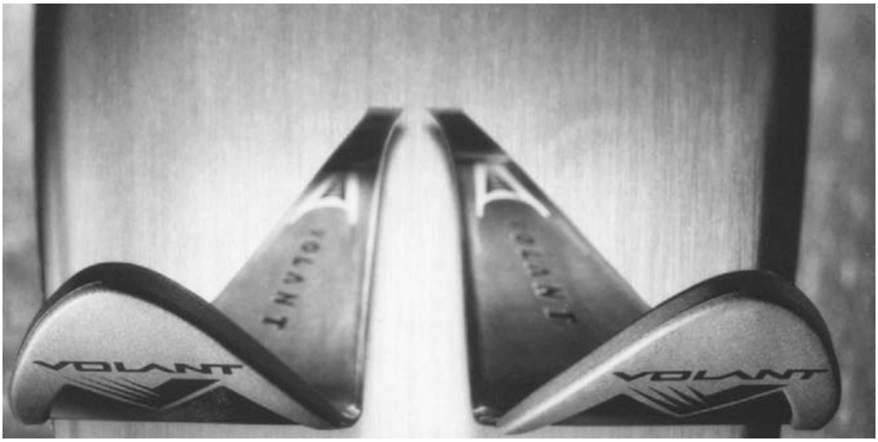

Fig. 19 Volant titanium skis.

should the line be drawn? Or should there be no restrictions? The solution, according to a young man in one of my classes at the University of Idaho, is that if you have the patent, you (and only you) can use this material or design no matter what it is.

This then brings us to a second question. Should we allow competition at the highest level to be only affordable to the elite because of the high cost of equipment? In turn this leads into what is perhaps the most controversial question. If we allow certain classes of materials and designs, but not others, we can actually favor the class of people who will excel! Let us briefly examine these questions.

The carbon fiber vaulting pole, javelins with spiral tails, golf balls with special dimple patterns, stiffer carbon fiber tennis rackets, bicycles with new types of wheels, and the "egg position," discuses with their weight distributed as close as possible to their perimeter, and America's Cup yachts (forget it, this one is too complex) all lead to "further and faster." We should not be Luddites but where should this end? Can we ensure that it is people who are competing and not the advanced materials? For sure, we do not want to force the paraplegic athlete to go back to a wooden pylon. But how about electronically guided darts, heat-seeking missiles for grouse shooting (allowable, provided the grouse is still edible, according to a young lady in one of my classes at the University of Idaho), solar-energy-enhanced bicycles, and terrain-following golf balls, which automatically find the lowest local elevation on a putting surface (the bottom of the hole).

Returning to running, consider the advanced devices that allow the runner to be catapulted forward more efficiently than if he were running on two human feet (cost up to $7000). So let's develop similar devices to be incorporated into the shoes of Donovan Bailey (winner of the 100 m in Atlanta in a world record 9.84 s) and Michael Johnson (winner of the 200 m in Atlanta in a world record of 19.32 s). Any bets on whether Donovan and Michael could then break 9 s and 18 s in their respective events? Is the answer here that nothing beyond the "natural" springiness should be allowed?

What is the answer to high costs? Many sports rules committees feel that a reasonable compromise is to keep their sport affordable to many athletes rather than the elite few. For example, disk wheels were initially banned from Olympic bicycling competition because they were so expensive that they could not be considered as available to most cyclists. Should all canoes and kayaks conform to a single-hull design? Should the "Hoyt bow," comprised of tiny glass beads embedded in a rigid foam matrix, be allowed to compete against the traditional wooden bow? The lack of change in behavior with temperature and minimal moisture absorption of the new bow has led to winning scores of 1350 (out of 1440) compared to about 1100 thirty years ago—but at a price.

Let us take two examples of how legislation regarding advanced materials can significantly determine who wins an event. Elite rowers stand 6 ft 6 in. and weigh about 210 lb. In contrast elite kayakers stand 6–12 in. smaller and weigh 30 lb less. This relates to allowable boat dimensions—a rowing boat can be as long as desired, favoring strength even at higher weights (longer boats can distribute more weight over a larger area without riding too low in the water, which leads to excessive drag). In contrast the length of a kayak is restricted, leading to an optimized aerobic strength-to-weight ratio.

When the javelin was redesigned to be lighter and more aerodynamic, finesse was required to make it "float" correctly. Masters of this new art, such as Uwetlohn of East Germany (1984), deftly projected the javelin over 100 m, much to the danger of spectators and other athletics warming up on the far side of the stadium. The 1986 ban on the new design led to a dramatic drop in the world record by 20 m, with the "finesse floater" fading to the second rank, and the "strong-arm boys" once again leading the way.

Lots of questions, few answers.

5 CONCLUDING REMARKS

In this chapter, I have chosen a number of sporting events to illustrate how the magnitude of the effect of advanced materials differs depending upon the sport in question. I then presented some ethical issues regarding the use of advanced materials in sports, with the issues outnumbering the answers. Clearly different sports have been affected by quite different amounts, in some cases to an extent that has required legislation restricting certain materials and designs (titanium baseball bats, floating javelins, nonconforming golf club drivers, etc.). However, perhaps the best approach would be to define acceptable performance standards rather than the conventional ad hoc regulation focused on design standards.[11] This should protect the essential skills of the sport, preventing it from becoming too easy or distancing it from historic or aesthetic origins. For further details on the use of advanced materials in sports, the interested reader is referred to two recent conference proceedings.[21,22]

Acknowledgments

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CHAPTER 41

MATERIALS SELECTION FOR WEAR RESISTANCE

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1 INTRODUCTION

The selection of materials and methods for wear applications is an important part of both technological advancement and manufacturing activities. However, materials selection is often viewed as a random process or worse. The individuals charged with designing new parts, developing new processes, or overseeing component trade study projects rarely have had the opportunity or time needed to develop a "feel" for the general materials performance of metals, ceramics, or plastics during a typical undergraduate university education. The good news is that ignorance is curable and its treatment should leave no permanent scars. Methods and approaches to solving materials problems have been developed over time that may help clarify needs and reduce the degree to which materials selection may be considered a "black art." Materials application, performance, and manufacturability are all key parts in the selection for wear resistance applications, but the general methods are also extensible to other areas of materials selection.

There is a great deal of interest in replacing hard metallurgical coatings with materials and systems that are more environmentally benign and are capable of providing equal or better performance than those materials they replace. Materials replacement efforts have traditionally relied on the shotgun approach where a material is simply substituted for another. This particular approach, however,

Handbook of Materials Selection. Edited by Myer Kutz

rarely ensures success. Numerous factors must be taken into account when choosing a replacement for a hard coating. These factors include the temperature, work face pressure, chemical environment, materials compatibility, elastic constants, and cost. If more environmentally benign materials were easily substituted for traditional hard materials, then they would have been long ago. Because there is no direct substitution, it is necessary to tailor the replacement materials to the specific application.

The main tasks in materials selection for wear application are to first specify performance needs and then financial needs. The order of these activities is important because, while a lower cost component part may be preferred, that less expensive part's performance must be suited to the task. It is very difficult to specify a part mainly on cost without knowing the performance design limits. This chapter is concerned mainly with the issue of performance, but one always needs to be aware of the cost of component acquisition or manufacture.

The information base and deposition techniques developed for one class of materials can typically be extended to other materials in the same class. Examples of this include hard coatings such as silicon carbide and titanium nitride, soft coatings such as silver and gold, hardened metal alloys, and polycrystalline ceramics. General wear surface selection methodology for one class of materials may be extended to other unrelated classes.

2 PROPERTIES OF WEAR MATERIALS

A wear material may be used to reduce dimensional changes due to unwanted material removal, reduce frictional losses, to tailor the physical performance of a component, and/or to provide a physically stable working surface. Wear can be divided into several categories such as adhesive and abrasive wear that take place during sliding contact. Surface fatigue and deformation wear are an impact or loading rate phenomenon, and corrosive wear is caused by the interaction of the wear surface with the local environment. These wear mechanisms may act singly or in combination with one another to alter a surface. The proper selection of a material for a wear application will strongly depend on both the type of wear to be countered and on the wear environment. The wear environment can be dry, wet, warm, cold, and so on. Wear taking place in a corrosive marine environment will be more damaging than the marine environment or the wear alone. Wear phenomenon is a factor in applications where it might not be readily apparent. Optical windows that are exposed to the natural elements have a need for wear protection where dust, sand, and ice can impact and roughen soft optical surfaces. Fan and propeller blades in water can experience wear by cavitation erosion in water and bug and dust impact erosion in air.

3 MATERIALS SELECTION PROCESS

The classic method of selecting a wear material is to use what has always been used in the past for a particular application. There is a reason for this—it works. For example, steel ball bearings are relatively inexpensive and are superb at what they do *if* they are not pushed beyond their performance limits. A good reason would be needed to replace steel ball bearings in an application for an alternative material or a different type of bearing. Changes in performance needs such as increased rotational speeds, a need for mass or volume reduction, altered mechanical shock environment, or increased reliability could lead to a demand for an alternative material.

Few particulars are provided here in terms of specific wear materials selection. Each wear application needs to be approached as a unique situation if a "best" result is to be achieved. That said, the following guidelines can allow for the rapid selection and insertion of an optimal wear system into operational use.

1. Specify the maximum operational limits of the wear materials system for safety and lifetime. Properties that make some wear films excellent for one particular application may be completely unsuitable for other uses.

2. Specify the normal operational parameters and acceptable performance criteria of the wear materials system. Performance criteria would include the number of cycles of use and the physical and chemical exposure environment before, during, and after use. This step provides for the selection of a broad range of materials and technologies that could fit the needs of the application. No preemptive elimination of technology should be attempted at this stage. Some of the materials and technologies may later be found to be mutually exclusive or inappropriate for use at a later point. Preemptive preselection at this point may eliminate "poor" candidates but also serves to too narrowly focus the materials search too early in the process. Early candidate elimination is attractive, but it can eliminate an entire class of potential solutions and can possibly restrict the ultimate wear material selection to a good solution but perhaps not the best solution. The more care that is taken during this crucial step will enable the actual materials selection phase to be much smoother in terms of performance, availability, and price.

3. Establish the degree of mechanical, physical (thermal expansion, dielectric constant, and so on), and chemical compatibility the wear material must have with the system. The real process of wear material selection begins once the preceding steps have been taken.

4. Material availability and cost are closely related factors usually taken into account at the same time. The cost of a particular wear material is almost exclusively controlled by its availability. Availability is directly controlled by prevalence of use (numerous examples exist of high-priced finished parts made from more common materials than their lower cost cousins) with the attendant savings of resulting from high-volume manufacturing. Availability of raw materials and the ability to work, shape, and form those materials can also influence materials cost.

Cubic boron nitride is an extremely hard wear material used in tooling for ferrous metals where diamond is inappropriate. It is a completely synthetic material not found in nature and is produced mainly as a bulk powder in high-pressure growth apparatus in limited batches. The powder is then used as a loose abrasive or it can be reworked into a solid piece of tooling. For all practical purposes, cubic boron nitride is unavailable as a directly applied coating.

The need to process the material after its synthesis adds yet another layer of expense to the cost of tooling. Cubic boron nitride tends to be expensive due to its availability. Titanium nitride is a fairly hard materials that is also completely synthetic but it is easy to produce. Inexpensive titanium-nitride-coated tooling is now available from many suppliers.

A General Hierarchy in Cost of Manufacture of Wear Materials

Bulk materials of commonplace composition that are easy to work and form

- **(a)** Materials that can be made in final shape with no post processing
- **(b)** Materials that can be made wear resistant after final shaping as by tempering of metal or firing a ceramic

Coatings of commonplace composition that may be applied to easily manufactured substrates

- **(a)** Coating applied under ambient conditions such as room temperature and pressure
- **(b)** Coatings formed in nontoxic water baths
- **(c)** Coatings and treatments that require high temperatures and controlled atmospheres
- **(d)** Small batch vacuum-based treatments

Wear materials that are formed *ex situ* and then are attached to a substrate

- **(a)** Gluing
- **(b)** Cementing
- **(c)** Brazing
- **(d)** Diffusion bonding

Bulk materials of uncommon composition that are difficult to work and form such as solid carbides, borides, and silicides

4 MANUFACTURING PROCESS SELECTION

Process selection is a second-tier consideration in most instances of wear materials selection. The physical properties of wear coatings may vary depending on the deposition method and technique. The standard cost savings from continuous and semicontinuous manufacturing methods such as extrusion and rolling versus stamping and milling operations also apply for wear materials. Method of manufacture becomes very important when directional or textural property characteristics of a material need to be considered. Many of the physical, optical, chemical, electrical properties of wear films will be controlled or modified by their degree of deviation from perfection imparted during manufacture. This is a common consideration in the area of composites manufacture. The component materials of a composite structure are only slightly more important than the manner in which they are arranged in space and bound to one another. Workpieces with apparently similar material compositions can have dramatically varied performance characteristics depending on the arrangement in space of their component parts as influenced by their method of manufacture. The physical properties of wear films can be modified and controlled by altering deposition parameters, using additives, changing substrate selection, and varying the seeding and nucleation method.

5 BASICS OF WEAR MATERIALS

Wear materials tend to be one of two types: (1) bulk solids or (2) coatings, films, and surface treatments. A bulk wear material will typically provide long-term wear surface use. There is more of a wear part available for continued use provided that significant dimensional changes have not affected the actual performance of the tool. However, the lack of availability of a wear material in bulk form or difficulty or expense in its manufacture can force the use of coatings or films instead. Likewise, coatings or films can enable the use of a material that is unsuitable in the bulk form but has very good performance when combined with other materials in a multicomponent wear system. The trade-off in the use of bulk and film materials is the possibility of enhanced performance of a coated tool versus the cost and effort required to make the coated tool. Other factors such as increased tool lifetime and length of time between tool changes may make a coated wear system attractive with respect to an inexpensive bulk material system. Cost and availability are two factors that strongly govern the selection of a bulk material or a surface treatment for a particular wear application.

The following references provide a rich resource for review and further exploration of the properties and behavior of wear materials: Buckley and Rabinowicz (1986), Apachitei and Duszczyk (2000), Bull and Matthews (1992), Bull et al. (1988), Formanek et al. (1993), Jackson and Mills (2000), Joost and Schwedes (1996), Karja et al. (1993), Foroulis (1984), Dobrzanski (2001), John (1984).

The general application of wear materials in varied situations are addressed in a number of the following references: Ball and Ward (1985), Balon and Aizinbud (1989), Berns (1995), Bull et al. (2000), Burkle et al. (1995), Cooper et al. (1992), Franklin and Beuger (1992), Franklin and Dijkman (1995), Gagg (2001), Gandhi and Agrawal (1994), Garbar (1995), Gates and Eaton (1993), Geiger (1992), Hsu et al. (1991), Hsu and Shen (1996), Jang and Kim (1996), Jilbert and Field (1998), Jones (1997), Jung (2000), Klocke and Krieg (1999), Korsunsky et al. (1995), Kuljanic (1992), Kurzynski (1996), Larsen-Basse (1990), Lempert (1988), Llewellyn (1996), Lohmann and Van Valkenhoef (1989), Lyons (1998), Mainwaring (1994), Manning et al. (1984), Margus and Comerford (1994), Martinella (1993), Medley (1992), Meyerrodenbeck et al. (1992), Mikhailin et al. (1985), Nuttall (1985), Onate et al. (1998), Paller (1991), Pascheto and Behnood (1997), Pejryd et al. (1995), Penlington et al. (1995), Phillips and Knapp (1995), Ramalingam and Zheng (1995), Reinhard and Volz (1983), Robinson et al. (1993), Rozenberg et al. (1987), Sare and Arnold (1995), Sessler et al. (1993), Sexton et al. (2001), Stack (1997), Stewart (1997), Stokes and Cooley (1985), Suchanek et al. (1999), Thompson (1994), Uma Devi and Mohanty (1998), Voronenko (1992), Ward et al. (1996a), Wassell et al. (1997), and Wendl and Wupper (1991).

Hardness is related to wear in that if it is very difficult to break one bond, then the chances of breaking many bonds (wear) will be low. A related series of references deal with the use of hard materials in wear applications: Bulloch and Henderson (1991), Beck et al. (1993), Knotek and Loffler (1992), Knotek and Loffler (1991), Zahner and Menon (1995), Williamson and Bolton (1983). The selection of specific wear materials for specific applications is reviewed in Ashby (1992), Bolvari and Glenn (1995), Bamkin and Piearcey (1990), Charles et al. (1997), Carnes et al. (2000), Edwards (1994), Edwards (1997), Eyre (1991), Farrow and Gleave (1983), Fischer (1996), Sundaresan (1988), Syan (1994), Strafford (1996), Subramanian and Strafford (1993), Subramanian et al. (1996), Shubrook (1996), Glaeser (1992), Hogmark et al. (2000).

6 SUBSTRATE SELECTION

There are several basic principles involved in substrate selection for wear film deposition. The material has to be compatible with the chemistry of the wear film deposition and growth environment. The substrate cannot have a coefficient of thermal expansion that is far greater or less than that of the wear film. This criterion can be reduced in importance by the use of very thin films that tend not to build up large internal stresses.

7 SURFACE MODIFICATIONS

The as-grown surfaces of some hard films are not suited for immediate use. The as-formed surface of some wear materials can be very rough. The degree of roughness can prevent the use of these materials as bearings if there is no method of making the surface smooth. These surfaces may be rough or chemically reactive and require an additional preparative step such as polishing or a “run in” period prior to their use.

Films need to have less than a 0.4-μm peak-to-valley roughness in general to be used for bearings. Surfaces with roughness greater than 5 μm peak-to-valley roughness have been found to be unacceptable and, in the absence of a method of making the surface smooth, would prevent the use of these materials as bearings. Mechanical polishing is preferably avoided to reduce the per-part finishing cost as well as retain uniform dimensionality.

Making a wear film smooth to begin with reduces the likelihood of introducing flaws into the film as well as making the process much less expensive. Smooth wear films may be made by several different methods. These include polishing, brazing the rough side down, growing the films very thin, or growing them very smooth initially. Smooth films can be made if the average crystallite size is small and if there is no room for the crystal to grow laterally. A thin, pinhole-free film can be grown if crystallites are densely packed. Making the film smooth initially reduces the likelihood of introducing flaws into the film as well as making the process much less expensive.

8 FILM THICKNESS

A thin film will allow the physical properties of the substrate material to be sampled during use. The scale of thickness is completely dependent on the scale of the system. A thin film might be 0.5 μm thick if the maximum foreign particle size is 0.25 μm in diameter where a thick film would be 5 μm thick. Similarly, a thin film might be 70 μm thick if the maximum foreign particle size is 20 μm in diameter where a thick film would be 200 μm thick. How thick must a film be to be thick enough? The size and hardness of foreign particulate matter arriving at the bearing surface will determine the thickness of the wear film. Thickness removal is comparable to cost because of time and material used during finishing. Mechanical polishing can also be an agent of flaw introduction. A brittle hard coating that is only a few microns thick would easily run a through-crack.

9 APPLICATIONS AND EXAMPLES OF WEAR MATERIALS

Wear materials are generally thought of in terms of metallurgical materials systems. Overall volume of wear materials would certainly demonstrate the importance of metals and metallurgy. There are a variety of other materials that have and are being used in wear applications. While the total volume of these materials combined is small compared to metals, they do represent a significant fraction of wear materials. Ceramics are slowly being phased in as wear materials in expected and unexpected places. Ceramics are now being used in high-performance ball bearing applications as well as high-end cutlery. The studies by Dellacorte and Steinetz (1994) and Riley (1996) review some of the uses and methods of selection of ceramics for wear applications.

Polymers have had a traditional role in wear applications from Teflon bearing sleeves to the rubber tires strapped to the sides of harbor tugboats. The following works provide some general guidelines for polymer use and selection in wear applications: An et al. (1997), Besic (1995), Palmese and Chawalwala (1996), Leger (1989), Price (1987), Wolpers and Hager (1990), Sysoev et al. (1986), Sladkov et al. (1998), and Tewari and Bijwe (1991).

Stone and natural glass were some of the original wear materials used by humans. These materials continue to be used into the twenty-first century in “cutting edge” applications as described by Twitchen et al. (1995) and Ertingshausen (1985).

Injection, stamping, and figure mold surfaces in materials process facilities face significant wear problems. This type of machinery will lose its dimensional tolerance over time as it is used. An intricately carved stamping blank would ideally never change its dimensions. This would reduce long-term production costs and reduce the amount of mechanical downtime when workers are idled while waiting for a stamping press to be retooled. The following studies examine wear materials for this type of application: Clarke (1985), Elfick et al. (1999), Haggag (1989), Hu et al. (1999), Murray et al. (1997), Bahadur (1993), Ward et al. (1996b, 1998), Hampson (1994), Gonzalez et al. (1999), Atkinson and Bristol (1992), Aksit and Tichy (1998), Stack and Pungiwat (1998), and Haugen et al. (1995).

The purpose of numerous refernces cited above is to provide a resource to those interested in wear materials selection. References cited range from wear materials selection criteria to interesting accounts and analyses of wear materials application and failure. The following references may also be of interest: Blau and Gardner (1996), Collins (1981), Colombie et al. (1987), Freimanis et al. (2000), Fu et al. (2000), Gil Sevillano (1997), Hepp et al. (1997), Hornbogen and Schafer (1981), Middleton and Coupland (1996), Moore (1981), O’Brien (1982), Peterson and Ramalingam (1981), Samuels et al. (1981), and Wendl and Wupper (1990).

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CHAPTER 42

DIAMOND FILMS

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1 HISTORICAL BACKGROUND 1287
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1 HISTORICAL BACKGROUND

The formation of diamond by artificial means has been an on-going effort for much of the last 150 years. Hannay, Moissan, and Parsons all attempted to make diamond artificially during the nineteenth century. Their claims have been repeatedly examined and usually dismissed. Many of the early attempts at diamond growth did not and still do not fall within the confines of scientific dogma with regard to high-pressure and temperature diamond synthesis; however, some of those early efforts are remarkably similar to those used today to make diamond at low pressures. A review of these early efforts can be found in the book by Davies (1984). Figure 1 presents a rough timetable to show the activity involved in diamond synthesis by chemical vapor deposition (CVD).

One early effort at diamond synthesis by Willard J. Hershey in the 1920s to the 1940s is usually overlooked. He claimed to have made diamond from excess carbon dissolved in molten iron. According to Hershey, the diamond formation took place during the rapid freezing of the molten iron mass. The resulting rapid shrinkage of the iron was supposed to produce a large compressive force on the carbon and convert it to diamond (Hershey, 1929, 1937; Hershey and Hamersley, 1940). Empirical observation had shown that natural diamond found in its native kimberlite was probably made deep in the earth and the carbon that diamond was made from was under a very high temperature and a large pressure at the time of formation (Bauer, 1968). It was believed that diamonds could be made synthetically if the very high pressures deep in the earth could be replicated at a high temperature in the laboratory. However, attempts to make diamond in ways that did not incorporate the use of large hydraulic presses to create high pressures such as Hershey's were not looked on favorably. P. W. Bridgeman

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Handbook of Materials Selection. Edited by Myer Kutz

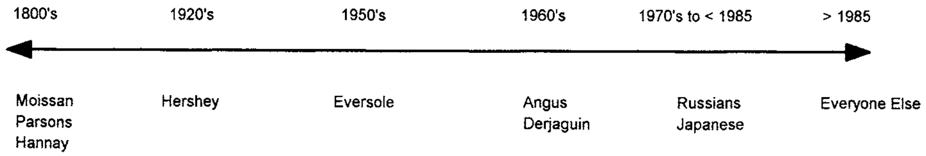

Fig. 1 Time line for the development of CVD diamond.

worked for many years on the production of high pressures in the laboratory as well as on the high-pressure graphite-to-diamond transformation (Bridgman, 1913, 1947) and could be considered the spiritual father of high-pressure and temperature synthesis of diamond. General Electric was the first to publicly announce that artificial production of diamond had taken place in 1952 via the high-pressure–high-temperature press method (Bundy et al., 1955; Strong, 1989) although ASEA in Sweden was the first to actually achieve high-pressure diamond synthesis (Liander, 1980).

The first, generally acknowledged, successful low-pressure diamond growth was announced by Eversole in 1962. His work had begun in the late 1940s, but he waited for conclusive proof of diamond formation before patenting his technique (Eversole, 1962a, 1962b). He made diamond by the pyrolysis of methane at low pressure over a diamond substrate and subsequently removing the nondiamond carbon to produce a net deposit of diamond. This was a *very* slow and laborious process and required the measurement of very small weight changes in the diamond. The amounts of diamond were so small that very precise measurements of less than one percent of weight change were needed to characterize the deposition process. This work was performed well before the development of laser-based Raman spectroscopy, the most common laboratory technique used to identify diamonds since the late 1980s (Knight and White, 1989). Eversole's work was reported shortly after the efforts of General Electric captured newspaper headlines around the world and was summarily ignored. Eversole's work may be taken as the historic beginning of today's efforts in the low-pressure CVD diamond synthesis and development.

A Soviet research effort was begun in parallel with Eversole in the 1950s on the pyrolysis of hydrocarbons over diamond substrates, but this early work was not reported in the western literature until recently (Spitsyn, 1991). J. C. Angus of Case Western Reserve University took up work on diamond deposition from the gas phase in the late 1960s (Angus et al., 1968) and apparently inspired the active Soviet research group to redouble their efforts on low-pressure diamond synthesis (Derjaguin et al., 1968, 1973). Low-pressure diamond synthesis was dealt nearly a death blow when Derjaguin was found to have produced a case study in pathological science with the announcement of polywater. Angus has said in public that this had the effect of reducing or removing any support he had for low-pressure diamond development, and he switched to pursue research on diamondlike carbon (Angus, 1986). Work on low-pressure diamond formation by the Soviets did not stop as a result of polywater. They continued to work through the 1970s on many aspects of low-pressure diamond deposition (Spitsyn, 1991).

The Japanese have also been working in the area of carbon film deposition, diamond-like carbon, and diamond since the 1960s. Based on their Japanese patent activity, their early efforts appear to mainly have been in the area of films for electronic materials (Tither et al., 1989). The Japanese were the source of three fundamental improvements in low-pressure diamond synthesis. They seem to have been the first to add large amounts of hydrogen to the deposition environment. Earlier low-pressure diamond deposition experiments were performed in environments containing mainly the hydrocarbon feedstock. After a deposition, the diamond substrate would be cleaned of the nondiamond carbon that would deposit with the diamond. The diamond substrates did not have to be removed and cleaned after each pyrolysis by depositing diamond films in a hydrogen-rich environment. This dramatically improved the growth rate of the film process. The Japanese were also the first to explore diamond film deposition with microwave plasmas, radio frequency plasmas, combustion torches, and hot filaments. Microwave plasma-enhanced chemical vapor deposition (MPECVD) is now the standard by which all other deposition techniques are judged. The third improvement was their introduction of Dr. Rustum Roy to diamond film synthesis in 1985 when he visited a series of Japanese laboratories to explore cooperative research.

Dr. Roy's return to the United States with news of the Japanese making diamond films prompted a strong American research response to either confirm or disprove the Japanese work (Roy, 1987). Since that time, over half a dozen companies had been formed to exploit diamond film technology and a industrially funded university consortium of 40 companies had formed within 2 years of his return. Activity in the field of diamond film deposition peaked around 1990. Six international conferences took place, several industrial and university centers had been founded for the purpose of diamond research, and a peer-reviewed journal devoted to the subject was initiated that year. A monthly news magazine (*Diamond Deposition: Science and Technology*) with hundreds of subscribers had been founded by that time to keep researchers up to date on the fast changes occurring on nearly a daily basis in the field. Today, the frenzied pace of activity in the field has slowed as research on diamond films moves from a part-time laboratory curiosity to an applied development field where useful products are gradually being produced (Fig. 2).

2 PROPERTIES OF CVD DIAMOND

CVD diamond may be used in thin-film form for mechanically hard infrared transmission coatings, machine tooling, electronic devices and substrates, and coatings for bearings. Thick diamond films may also be used in machine tooling but also as free-standing optical and infrared windows, electronic substrates, and bearings. CVD diamond may be made in powder form that can be used as the starting material for diamond ceramics and diamond filled composites, monosized abrasives, and as feedstock for high-pressure diamond crystal synthesis (Spear, 1989).

The physical properties of diamond may vary depending on the deposition method and technique. The general physical properties of diamond films are closely related to those established for bulk diamond. Many physical phenomena depend on the type of bonding and the bond termination that is found in a

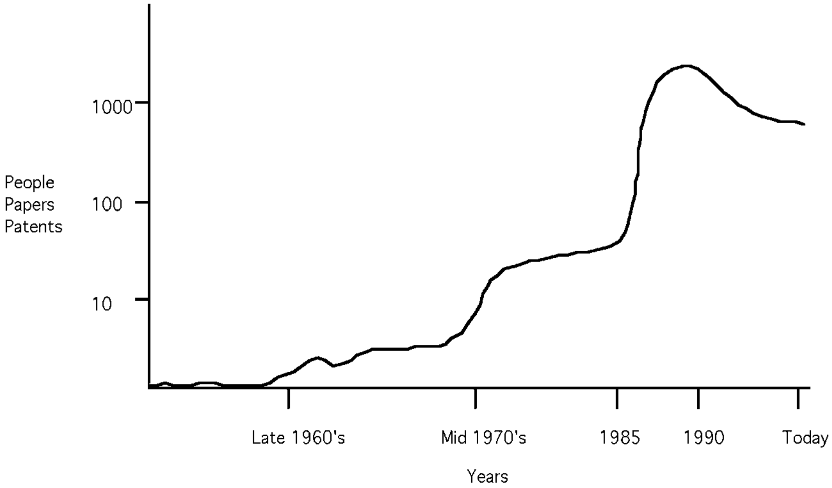

Fig. 2 Research activity in CVD diamond during the last 30 years.

material. Many of the physical, optical, chemical, and electrical properties of CVD diamond films will be controlled or modified by their degree of deviation from perfection. Imperfections may take on many forms. They can be structural, such as grain boundaries or polytypes; chemical, such as variations in stoichiometry; or optical, such as variations in refractive index or optical handedness. There are also less obvious imperfections such as disordered ferroelectric domains, irregularly distributed isotopes, or interfaces between discrete uniform regions of the same or different materials (Davies, 1994).

Diamond is the hardest naturally occurring material (Field, 1991). The great hardness of diamond makes it appropriate to consider diamond as a low-wear material. If it is very difficult to break a bond then the chances of breaking bonds (wear) will be low. Several other materials have higher bond strengths than does diamond, but those strengths are anisotropic. The hardest material that is known is a synthetically prepared type of diamond. Analysis of elastic constant data for ^{13}C diamond has shown that its bond strength is greater than that of ordinary natural diamond or commonly made CVD diamond. The reason for this is the slightly shorter bond length that results from having an extra neutron in the carbon atom's nucleus. Actual hardness measurements have not been made on the material to date, but the strength should scale with the elastic constants (Ramdas et al., 1993). Additionally, diamond has a low coefficient of friction (0.01) in the presence of air or hydrogen (Field, 1991). This makes the material as slippery as Teflon but far more wear resistant. The slippery nature of the hydrogen-saturated surface would reduce both frictional horsepower losses and decrease surface erosion when used in wear applications (Phelps, 1993).

The thermal conductivity of diamond is a good example of the structure–property relationship in CVD diamond. Annealed copper is the standard for thermal conduction in the area of high-power electronic components because it has a relatively high thermal conductivity of 4 W/cm K. Diamond is the best thermal conductor that exists. Diamond is very attractive material for use in electronic devices because a defect-free natural single-crystal diamond has a thermal conductivity of around 20 W/cm K (Seal, 1970; Seal and van Enckevort, 1988). An average polycrystalline CVD diamond film made under typical laboratory conditions has a thermal conductivity of 6–10 W/cm K (Plano and Adar, 1987). The values for polycrystalline CVD diamond are impressive until they are compared to isotopically pure single crystal diamond. Isotopically pure single-crystal diamond is made by taking polycrystalline isotopically pure diamond made by CVD and using it as the feedstock in single-diamond high-pressure synthesis. Recent measurements on that material have failed to find the upper limit on the thermal conductivity of the samples. The value currently being quoted is 36 W/cm K, nearly twice that of the best natural single crystal (Banholzer and Anthony, 1992). Thermal stress gradients caused by rubbing are not supported by diamond. Heat transfer in diamond is so fast that the temperature throughout the diamond film is always uniform and the film does not suffer from differential thermal contraction.

Diamond films are commonly considered to be excellent electrical insulators because of the 10^{15} Ω/cm resistivity measured for many natural diamonds. However, the resistivity of CVD diamond has been found to change with film composition. The electrical conductivity of CVD diamond has been shown to vary with the amount of hydrogen absorbed in the structure of the film. Ravi and Landstrass (1989) report that the resistivity of CVD diamond films can be correlated more with the amount of hydrogen they contain than with their Raman spectra. An "as-grown" diamond film had a resistivity of about 10^{6} Ω/cm. This same film was soaked for 30 min in a 600°C flowing nitrogen atmosphere, and the resistivity was found to increase to about 10^{13} Ω/cm. The process was found to be reversible, and the hydrogen could be put back into the sample, and the resistivity would decrease to near the as-grown value. Diamond films with significant amounts of graphitic structural bonding have been found to be electrically conductive and have physical properties that are a fraction of the natural properties, such as thermal conductivity. Figure 3 shows how many of the special properties of diamond lend themselves to specific applications. Several properties of diamond suggest an application in some cases such as with microwave electronic devices.

3 DIAMOND FILM DEPOSITION

Diamond films cannot be deposited on all materials and surfaces. Substrates that present problems to diamond deposition are the ferrous materials including iron, nickel, cobalt, in addition to copper, zinc, tin, and the precious metals. Diamond will grow on a substrate successfully at high temperature only if the material has a low solubility for carbon and forms a carbide. There is nearly always an interface that keeps diamond separated from its deposition substrate, and there is typically no direct diamond-to-metal contact. The interface usually contains at least a few atom layers of carbide (Srivatsa et al., 1989; Williams et al., 1990). As an example, titanium could support diamond growth through the formation of a titanium carbide interlayer. If, however, the carbide were too thick, then the difference in thermal expansion between the carbide and the diamond could fracture the interface. A diamond-like carbon film deposited on top of a substrate can also act as an interface instead of a carbide. Figure 4 outlines the elements

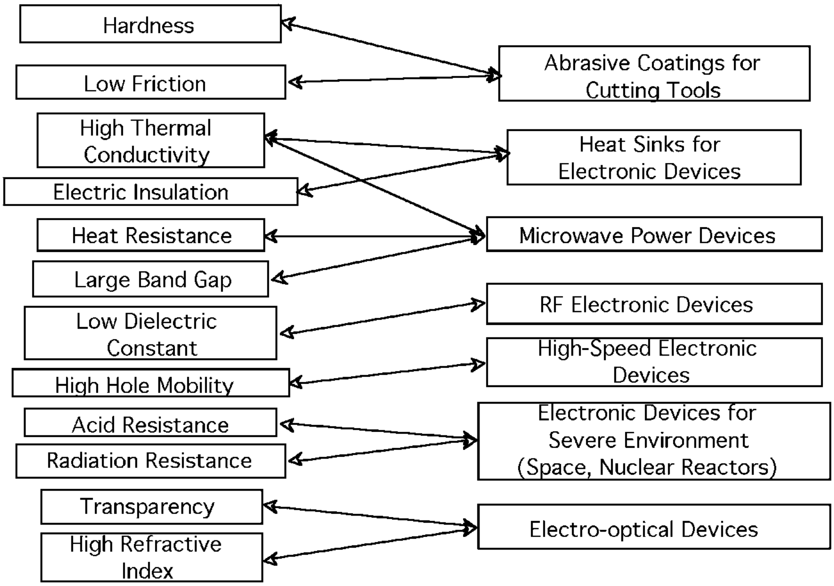

Fig. 3 Applications of diamond materials based on property criteria as adapted from Spitsyn (1991).

of the periodic table that are typically compatible with diamond growth either singly or in multiples. Phosphorus and molybdenum may only have a partial affinity for diamond growth. The other elements are more or less compatible with diamond growth.

Low-pressure CVD of diamond may be thought of as a process that is based on rate variations. The deposition and etch rates of carbon in the gas phase are

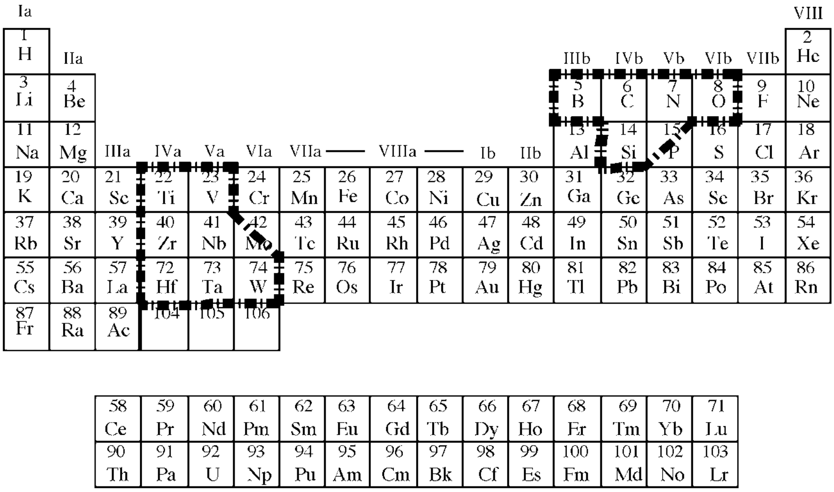

Fig. 4 Regions of the periodic table that show diamond deposition compatibility where the half boxes indicate partial compatibility.

the keystones in CVD diamond deposition. Carbon produced from the gas phase can take the form of many carbon materials. These carbon materials include diamond, graphite, diamondlike carbon (DLC), carbynes, fullerenes, soot, naphthalene, and so on. However, the rate at which each of these materials is formed depends on the total pressure of the system, substrate composition and temperature, gas temperature, flowrates, and feedstock. Most low-pressure carbon-producing systems produce nondiamond carbons in far greater abundance than they produce diamond. It is necessary to shift deposition parameters in a direction that favors diamond carbon growth at the expense of the growth of the other forms of carbon. However, this needs to be done while removing nondiamond carbon from the system. The presence of nondiamond carbons tend to poison diamond growth and interfere with the formation of good-quality diamond crystals.

The etch rate of diamond in hydrogen and oxygen appears to be far less than all other nondiamond carbons formed. This results in a diamond-rich carbon deposit. If diamond is being formed at 10% of the rate of the other carbon materials but is being etched at 1% of the rate of the other materials, then after a period of time a net deposit of diamond will form. Diamondlike carbon films or graphite will form if the substrate temperature is below 800°C. The substrate temperature must usually be somewhere between 800 and 1000°C during CVD diamond deposition. Diamond tends not to form if the substrate temperature rises above 1100°C. CVD diamond, single-crystal natural, and high-pressure synthetic diamond graphitize and etch above this temperature. Typically, CVD diamond forms in a very narrow temperature window. Deposition occurs when the methane plus hydrogen mixture is activated. Energy to activate the process may be in the form of radio frequency (RF), thermal (hot filament), microwave plasma, high-voltage direct current (DC), and others.

The selection of an appropriate diamond deposition process depends on the needs of the particular development program. Concerns about deposition rate, hydrogen concentration, substrate compatibility, and the ability to coat three-dimensional surfaces all should be taken into account. Three-dimensional CVD diamond films have been made by hot filament assisted CVD. Obata and Morimoto (1989) demonstrated the possibility of three-dimensional coatings by forming diamond hemispheres and sleeves. Their work used a filament-type reactor to produce these forms. Bachmann and Lydtin (1991) present a rudimentary chart to help in the selection of a deposition process presented here as Table 1. This type of chart should be viewed as a starting point from which the fine points of the assorted deposition techniques can then be compared for such things as cost, scalability, and ease of use as well as being capable of producing the particular type of diamond that is desired.

Properties that make CVD diamond films excellent for one particular application may be completely unsuitable for other uses. For example, the highest thermally conductive CVD diamond film has the least desirable physical properties for use as a hydrostatic bearing coating. Good thermal conductivity results from the material having a high degree of compositional purity and no nondiamond carbon in its structure. Nondiamond carbon has been found to help toughness and resiliency in CVD films. A low defect density is also necessary for a high thermal conductivity. Defects in the form of grain boundaries need to be

Table 1 Diamond Deposition Process Selection

	Rate (μm/h)	Area (cm^2)	Quality (Raman)	Substrates	Advantages	Drawbacks
Flame	30–100	<1	+++	Si, Mo, TiN	Simple	Area, stability
Heated filament	0.3–2	100	+++	Si, Mo, silica, etc.	Simple, large area	Contamination, stability, no O_2
DC discharge (low pressure)	<0.1	70	+	Si, Mo, silica, etc.	Simple, large area	Quality, rate
DC discharge (medium pressure)	20–250	<2	+++	Si, Mo alumina	Rate, quality	Area
DC plasma jet	930	<2	+++	Mo, Si	Highest rate, quality	Area, stability homogeneity
RF (low pressure)	<0.1	?	−/+	Si, Mo, silica BN, Ni	Scale-up	Quality, rate, contamination
RF (thermal, 1 atm)	180	3	+++	Mo	Rate	Area, stability, homogeneity
Microwave	1 (low pressure) 30 (high pressure)	40	+++	Si, Mo, silica WC, etc.	Quality, stability	Rate, area
ECR Microwave	0.1	<40	−/+	Si	Area, low pressure	Quality, rate, contamination

Source: From Bachmann and Lydtin (1991).

reduced for thermal conductivity, and so grain sizes must be very large. Large grains tend to have very high relief and produce rough films, also cracks are easier to run through large crystals than smaller polycrystalline masses. Adherent diamond films suited to hydrostatic bearings will need to be low in stress, tough, and probably not be as thermally conductive as single crystals.

4 MODIFYING CVD DIAMOND

The physical properties of CVD diamond can be modified and controlled by altering deposition parameters, using additives, changing substrate selection, and varying the seeding and nucleation method. For example, a carbon-rich environment will produce a diamond film with both a larger conductivity than natural diamond and one with a greater degree of compliance (Windischmann and Epps, 1990; Windischmann et al., 1991). Phelps and Koba (1989) showed that the electrical conductivity and the quality of their Raman spectra of boron-doped diamond films were found to increase and improve with increased diborane concentration in the deposition environment. Bernholc et al. (1988, 1992) predicted that with boron as a dopant “the reduced self-diffusion activation energy should lead to better quality material.” Phelps (1990) suggested that features observed in the Raman spectra of diamond films are the result of defects in the diamond structure, and boron doping of the material appears to substantially affect its Raman spectra. Defects in boron-doped CVD diamond have been shown many times to scale with the amount of boron doping in the material. A TEM study by Wang et al. (1992) showed cross-sectional views of several doped diamond at various dopant levels, and it is clear that the number of dislocations in the diamond structure show a marked decrease. The oxidation resistance of boron-doped CVD diamond is shown to increase with boron content by the temperature measured at the onset of oxidation (Loparev et al., 1984).

5 DIAMOND FILM ROUGHNESS

The as-grown surface of CVD diamond films is typically very rough. This degree of roughness (>5 μm peak to valley) would prevent the use of these materials as bearings if there were no way of making the surface smooth. This degree of roughness makes some diamond films very good at sanding and polishing rough areas on other diamond films. In order for diamond films to be used for bearings, they need to have less than a 0.4-μm peak-to-valley roughness. Surfaces with roughness greater than this have been found to be unacceptable for bearings made of other materials. Smooth CVD diamond films may be made by several different methods. These include polishing, brazing the rough side down, growing the films very thin, or simply growing them very smooth at first.

It is critical that a mechanical polishing procedure be avoided for smoothing diamond films. The reasons include the high per-part finishing cost. The only material that is known to successfully mechanically polish diamond well is diamond itself (Watermeyer, 1991). This results in an expensive raw material cost before any polishing activity even takes place. Also, a very thick film of diamond would have to be deposited so that some film would be left after the polishing procedure. Thickness is comparable to cost because of the slow deposition rates at which this material is deposited and the slow rate at which diamond is removed. A mechanical polishing step would also be the agent of flaw introduction. The diamond films made and likely to be used for bearing applications will only be a few microns thick. A crack could very easily run through the thickness of a diamond film during the polishing process because of diamond's brittle nature. Lastly, there is yet no method by which a surface coated with a diamond film with three-dimensional reentrant surfaces can be polished to a satisfactory smoothness while retaining size, shape, and surface quality specifications.

One technique that produces smooth diamond films is a flip-chip brazing technique. A rough diamond film is grown on a polished substrate and removed after deposition. The film may simply be turned over and brazed rough side down onto a second working substrate. The smooth interfacial surface is then the one that is outermost.

Smooth diamond films can be made by controlling the nucleation density and deposition parameters. Making films smooth to begin with reduces the chances of flaw introduction and makes the entire process much less expensive. Smooth diamond films can be deposited directly on the substrate growth surface with no postdeposition treatments. It was learned several years ago that thin, very smooth diamond films could be grown on polished tungsten through the use of diamondlike carbon films as an interlayer between the diamond and the substrate (Phelps and Koba, 1989). The diamondlike carbon deposition method will be covered in a later section on diamond film seeding techniques. The average size of a CVD diamond crystallite will be small if there is no room for it grow laterally. If crystallites are densely packed, then a thinner film can be grown that is pinhole free. A film will be smooth if it is very thin and densely packed. Large rough facets will not develop if crystallites can be kept small throughout the film thickness. This can be done by poisoning the diamond growth environment with excess carbon.

6 DIAMOND FILM THICKNESS

There are several aspects to deciding what thickness of diamond film is acceptable for a bearing surface. The working definition proposed by A. C. Gonzalez is that for a particular system "a thick film is one that will interact with the system as if it were bulk diamond" (Phelps et al., 1992).

In that same system, "a thin film will allow the properties of the substrate material to be sampled." The scale of thickness is completely dependent on the scale of the system. A thin film might be 0.5 μm thick if the maximum foreign particle size is 0.25 μm in diameter where a thick film would be 5 μm thick. Similarly, a thin film might be 70 μm thick if the maximum foreign particle size is 20 μm in diameter where a thick film would be 200 μm thick. A central concern is how thick must a film be to be thick enough. The size and hardness of foreign particulate matter arriving at the bearing surface will determine the thickness of the diamond film. Figure 5 shows schematically how the definitions of thick and thin apply to a sharp foreign object applied to the films under the same load.

A second concern dealing with thickness takes the deposition process and the mechanical system where the film will be used taken into account. Figure 6 shows the trades between having thick and thin films in a wear system. Currently, there is no correct answer to "what is the proper thickness?"

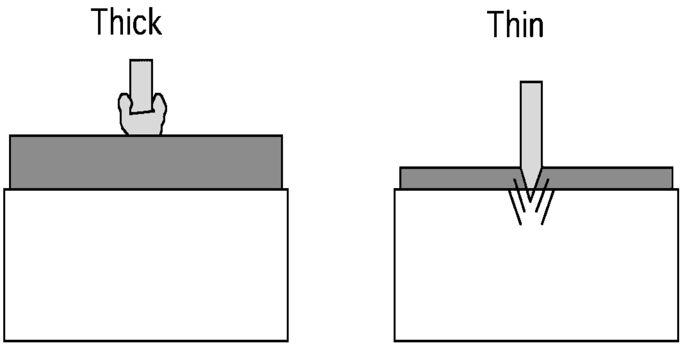

Fig. 5 Representing the difference between thick and thin coatings where a probe samples only the bulk film properties in a thick coating and a probe samples both the coating and the substrate in a thin coating.

7 DIAMOND FILM ADHESION

Adhesion of CVD diamond films to working substrates is one of the most important aspects of diamond wear coating technology. Diamond has the particular disadvantage that little is known about how to best attach a diamond film to a substrate surface. Good adhesion must be considered at the system design level in order to realize superior state-of-the-art bearings. Adequate adhesion between the diamond film and the substrate must be present in order to use the remarkable mechanical properties of diamond. There are many reasons why a diamond film would not be adherent. Identifying these reasons can lend direction to solving the adhesion problem. The first and foremost reason that diamond films delaminate from substrate surfaces is that the bond strength between the film and its substrate is not sufficient to hold onto the film. The strength necessary to hold

	Thick Films	Thin Films
Advantages	Longer wearing Bulk properties Reduce buckling	High intrinsic strength Lower residual stress Short deposition time
Disadvantages	Long deposition time High residual stress Film roughness	Film punch-through Short wear life Buckling

Fig. 6 Decision box showing relationship between thick and thin coatings.

onto a film will vary with the type of substrate, the film deposition conditions, the substrate preparation, etc. Means of increasing the bond strength between the diamond film and the substrate are chemical, physical, and mechanical. A chemical means of enhancing adhesion is through the use of carbide forming substrates. Molybdenum is frequently used as a substrate material for diamond deposition, however, diamond does not form a good strong carbide with this metal. A metal that is more suited to diamond growth is tungsten. Tungsten forms a strong, stoichiometric carbide that supports diamond nucleation and growth.

The thermal expansion of diamond is far less than all metals and many nonmetals. Most substrates that diamond is deposited onto have thermal expansion coefficients that are larger than diamond such as silicon and tungsten. Diamond films deposited on these substrates at 1000°C are in compression at room temperature. If the tensile stresses resulting from shear due to compression are greater than the bond strength between the diamond and its substrate, then the film will rupture at the substrate-to-film interface. The first means of controlling stress and, thus, adhesion would be to reduce the thermal expansion mismatch between the diamond film and the substrate. Pure silica glass has a lower degree of thermal expansion from room temperature to 1000°C than does diamond. Diamond is found to be in tension at room temperature after it has been deposited onto silica glass at 1000°C. However, the addition of a small amount of boron to the silica glass will raise its thermal expansion coefficient to near that of diamond. Interlayer materials such as diamondlike carbon or carbide formers such as tungsten or silicon could be used to control the thermal expansion difference between diamond and the substrate of choice. Likewise, a diamondlike carbon or carbide former deposited between the diamond films and the substrate could allow the use of substrates other than good carbide formers to be used for diamond deposition.

It may be possible to control and manipulate the residual stress found in deposited diamond films. Diamond films can be made more compliant by making the film very thin or by changing the elastic constants of the diamond film. This can be done very precisely by controlling the amount of nondiamond carbon that is present in the film. Adding diamondlike carbon or graphite to a diamond film reduces the stiffness of the material, and it should be possible to make a low-stress diamond film on tungsten. This could be done through a graded interface. A poor-quality diamond film would be deposited on top of the substrate, and during the deposition process it could be possible to reduce the nondiamond component and produce a good-quality diamond film.

The last means of enhancing the adhesion between the diamond film and its substrate does not lower the stress at the interface but increases the rupture strength of the interface. This is done by mechanically interlocking the film onto the substrate. The work of a team of researchers at the University of Minnesota (Tsai et al., 1992, 1993) has produced a method of depositing diamond films on incompatible substrates. This is done by electroplating nickel over a partially grown (island form) diamond film on a diamond-compatible substrate surface. CVD diamond is further deposited on top of and across the electrodeposited film and connecting the islands of earlier deposited diamond. The islands of earlier diamond act as attachment anchors. Later, a continuous diamond film is

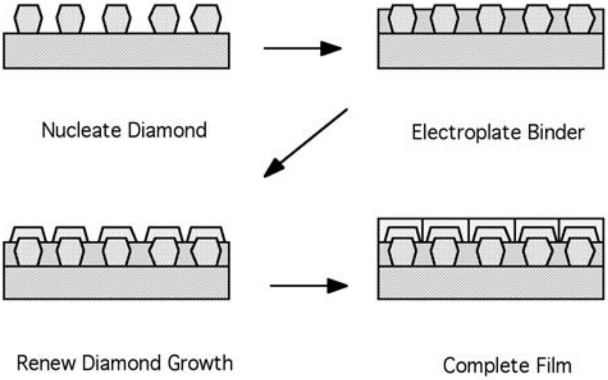

Fig. 7 Process steps used to electrodeposit a metal interlayer between the diamond film and the substrate.

grown across these islands. The substrate does not have to be compatible with diamond because no initial adhesion is necessary between the substrate and the diamond seeds. The substrate simply has to be compatible with the electrodeposited film on top and not form a eutectic composition at diamond deposition temperatures. This method holds great promise for depositing diamond directly on diamond-incompatible materials such as INCO 718. Adhesion is the single most important factor currently affecting the use of diamond films as wear and bearing surfaces.

There are several means by which an adherent diamond film may be produced on a substrate surface.

1. Deposit the diamond wear film on top of a strong carbide-forming substrate. This results in a strong diamond-to-carbide chemical bond and help hold the film onto the substrate.
2. Deposit the diamond film on an intermediate layer that is a strong carbide former deposited on top of the substrate. This results in a strong diamond-to-carbide chemical bond and a strong carbide-to-substrate chemical bond.

Film Adhesion	**Film Roughness**	**Film Thickness**
Controlled by:	Controlled by:	Controlled by:
Stress at Film Interface	Deposition	Deposition Time
Strength of Bond	Nucleation Density	Stress at Interface
Film Adhesion	**Film Roughness**	**Film Thickness**
Controls:	Controls:	Controls:
Maximum Film Thickness	Friction Losses	Debris Punch Through
Number of Restarts	Bearing Wear Rate	Service Life

Fig. 8 Relationships between film character, physical properties, and service behavior.

3. Electroplate diamond anchors onto the substrate surface (Fig. 7). This allows the adhesion of the diamond film to a substrate that has no chemical compatibility with the diamond such as steel or INCO 718.
4. Braze a free diamond film onto a prepared substrate. The brazing material is usually a low-temperature alloy that can be a copper–silver or other easily flowing alloy that contains powder of a carbide former such as titanium or zirconium. The carbide former makes a good chemical bond with the diamond surface and in turn bonds to the braze material also.

Figure 8 places the three critical aspects of diamond wear films into a context of their individual importance with respect to one another as well as how the threes aspects interact with the bearing system in general.

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CHAPTER 43

ADVANCED MATERIALS IN TELECOMMUNICATIONS

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1 INTRODUCTION TO COMMUNICATIONS

The communications revolution of the previous decade is based on transfer of information. Initially, most of the information transferred between people was via voice communication, that is, over the telephone network. Today, due to the birth of the Internet, the transfer of data now exceeds the network capacity demanded by voice communication even though the number of cellular phones is increasing dramatically and the cost per minute of long distance is decreasing. One may ask: Can the communication network meet this demand for capacity?

The capacity of any network, whether it is cable, wireless, or optical is related to bandwidth. Bandwidth literally describes the width of the frequency band, or window, that can be utilized to send information. Bandwidth is a limited resource. For wireless communication bandwidth is so valuable that it is regulated

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and sometimes sold by the government. Bandwidth in optical fiber now seems limitless, this is due to the small amount of information currently being carried along the fiber lines. Few people who are connected to the Internet utilize wide bandwidth services, such as Ethernet, cable modems, and digital subscriber lines. As more of these services are installed in businesses and neighborhoods, the demand for enormous data transfer rates will increase in the form of streaming audio and video. The maximum capacity of a network, which is the sum of all of the individual users' data transfer rates, is directly proportional to the bandwidth of the network.

Information can be transmitted in analog or digital format and with a wide variety of encoding techniques: modulation by amplitude (AM), frequency (FM), or phase (PM).[1] Regardless of the technique used to encode the information, the capacity of a network, also referred to as the data transfer rate, can be estimated by Shannon's equation,[2] where the capacity of the system, given in bits per second, is equal to the bandwidth in hertz for a system where the signal-to-noise ratio approaches 1 (i.e., the weak signal limit). Therefore, if the bandwidth is 1 MHz, then the capacity is equal to 1 million bits per second (1Mb/s).

The goal is to manufacture components that operate at high frequencies and over large frequency ranges in order to increase bandwidth. Based on these components, system engineers will be able to design high-capacity networks.

The current configuration of the communication network is illustrated schematically in Fig. 1. The three dominant communication networks are presented: cable, optical, and wireless. These networks are divided into local (metro, wireless) and long-distance (also known as long-haul) sections. Depending on the network design and transmission protocol (modulation, multiplexing, access), specifications are assigned for devices and components. Some devices are highlighted in Fig. 1, and their components are listed in the figure key. This illustration provides an overall view of the telecommunications business: markets, networks, devices, components, and materials. Envisioning dominant markets for the future and deciding which networks will evolve to meet capacity demands will aid in the determination of materials research directions.

This chapter will describe briefly the communication networks, illustrate in depth several key network components with attention paid to the materials used in these devices and the reasons for their selection, list some materials and their respective components, summarize various synthetic techniques for making these materials, and finally look toward the future for communications technology.

1.1 Networks

Cable

The original telephone network was initially assembled for use with telegraphs. Since then with the expansion of nearly a phone in every home, the telephone network links the world. This initial copper cable network was based on twisted wire pairs and coaxial cable. The twisted wire cables were assembled with 6–4200 pairs wrapped in a protective sheath either composed of polymer insulation with a copper core or a cost-effective alternative, an aluminum core.[3] Coaxial cable, pictured in Fig. 2, was used in the backbone of the wired network. A $\frac{1}{2}$-in. coaxial cable with 300 MHz bandwidth demonstrates 4 dB attenuation over

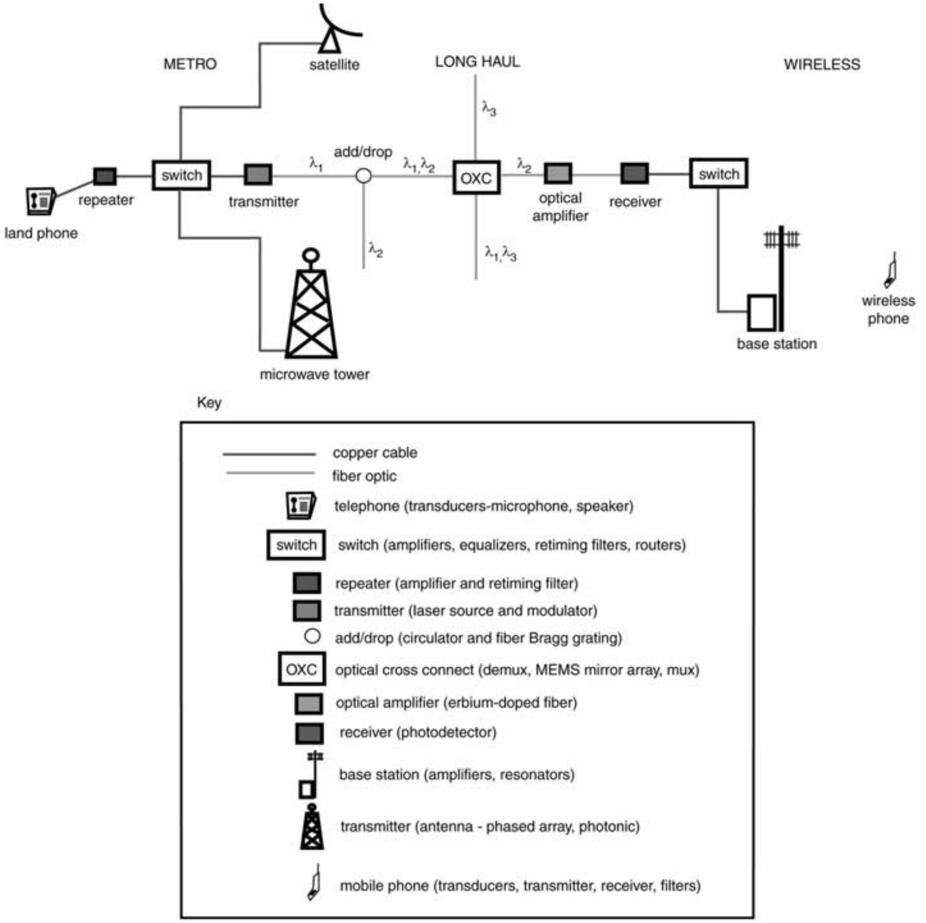

Fig. 1 Communication network.

100 m while operating at a center frequency of 300 MHz.[4] Coaxial cable has almost entirely been replaced by optical fiber and analog transmission was completely phased out by the year 1990.

Cables of twisted copper wire pairs still connect the ‘‘last mile’’ of the telecommunications network. The last mile refers to the distance from the end of the fiber-optic backbone to the home. Due to encoding techniques, the standard telephone network has a maximum data transfer rate of 56K bits/s even though the bandwidth of a twisted pair, which varies with the type of wire and its length, is 2 MHz over a one mile span.[1] Recently, this bandwidth limitation has been overcome by digital subscriber loop/line (DSL) technology.

Primary concerns of the copper network are loss (attenuation of the signal from resistance), crosstalk (mirror of another conversation through induction), and delay distortion (data transfer rate limitation due to capacitance of twisted wire pair). These are being addressed through an evolution toward optic fiber rather than incremental (and costly) advances to improve resistivity with silver

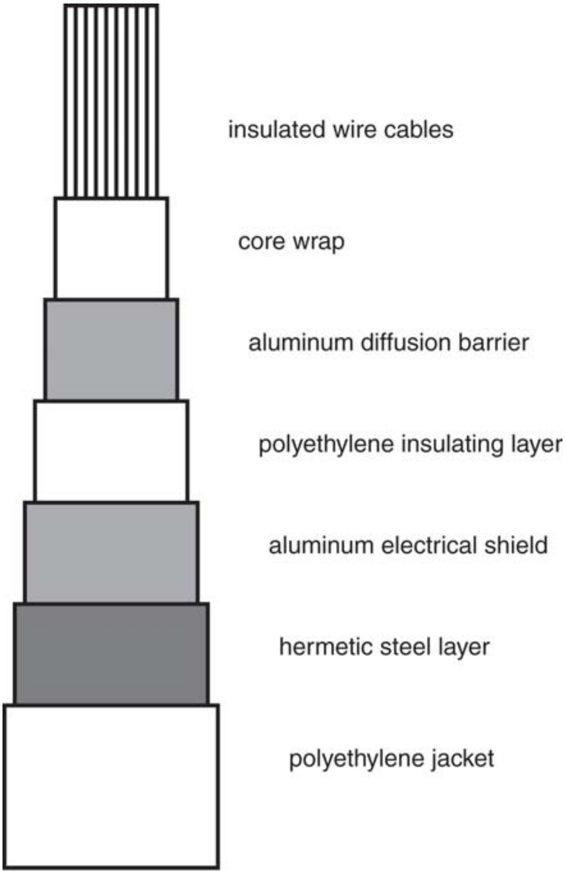

Fig. 2 Schematic of copper cable of twisted pairs showing layer structure and materials.

wire and lower dielectric constant insulation while still maintaining high breakdown fields.

Optical

Optical fiber possesses nearly infinite bandwidth, or so it seems. Currently, all Internet traffic can be carried on a single optical fiber. The only reason this is still true is due to slow modems using conventional phone networks, which are limited to below 56K (56,000 bits/s) data transfer rates, in addition to the limited number of people connected to the Internet at one time. However, this is changing daily. Cable modems and digital subscriber lines are now available in many areas in the United States yielding much higher data transfer rates up to 500 KBytes/s (500,000 bytes/s = 4,000,000 bits/s). In the near future, optical fiber to the home with extremely fast connections using transponders (optical modems) will greatly increase demand for capacity and thus bandwidth.

What is the maximum bandwidth of optical fiber? The usable wavelength range (related to frequency range) is from 1280 to 1620 nm (1.28 to 1.62 μm), a portion of the infrared electromagnetic spectrum. Restating this in terms of frequencies (2.34×10^{14} Hz to 1.85×10^{14} Hz) and taking the difference (0.49×10^{14} Hz) yields a maximum bandwidth of approximately 50 THz. Therefore, a conservative estimate of the capacity is roughly 50,000,000,000 bits/s, or 50 Tb/s. Recently, based on nonlinear effects in optical fiber, the maximum transmission rate is estimated at 100 Tb/s.[5]

This value for capacity is based on the acceptable wavelength range, which is determined by the transmission medium: silica fiber. Within this range, however, absorption due to hydroxyl (OH) groups in the SiO_2 fiber prohibits use within a band around 1.4 μm (Fig. 3).[6] In addition, losses due to Rayleigh scattering and vibrational absorption in the infrared place bounds on the wavelength range. For this reason, two main wavelength regions are used for telecommunication centered about 1.3 and 1.55 μm. The region at 1.55 μm is divided into three bands: C-band (common) from 1.53 to 1.56 μ, the L-band (long wavelengths) from 1.56 to 1.62 μm, and the S-band (short wavelengths) from 1.48 to 1.53 μm, which is commonly used for pump lasers. Further improvements have been made in fiber with the announcement of Lucent's Allwave fiber in which the hydroxyl concentration is negligible and therefore the entire wavelength range can be used to transmit data. Unfortunately, due to the absence of a broadband optical amplifier, the use of Allwave is limited to metro applications where repeaters are unnecessary. See Section 2.4 for more details on optical fiber.

The most flexible format with which to utilize the optical spectrum of fiber is wavelength division multiplexing (WDM). WDM is a type of frequency division multiplexing where each channel is separated in frequency by 200 GHz for coarse WDM or as small as 50 GHz for dense WDM (DWDM). Currently available optical networking systems can transmit data at 40 Gb/s per channel over 80 channels. Predictions indicate that systems with 320 channels at 80 Gb/s each are approaching quickly; that is a capacity of 25.6 Tb/s.[7] Perhaps the elegance of DWDM is the ability for each channel to be independently encoded.

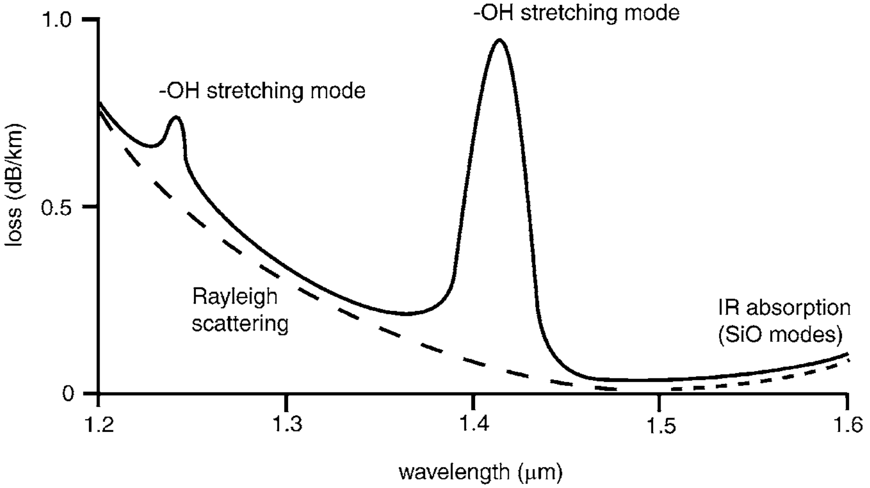

Fig. 3 Optical fiber transmission spectrum.

Therefore, a system can be upgraded sequentially such that some signals can be modulated at 2.5 Gb/s (currently employed networks) simultaneously with signal at 40 Gb/s. In fact, each channel can also be encoded via a different format, such as SONET, ATM, or IP.

Since optical communication has the benefit of incredibly high data transfer rates of terabits per second (Tbit/s), new encoding techniques were necessary to accommodate this bandwidth: SONET (synchronous optical network)/SDH (synchronous digital hierarchy) and ATM (asynchronous transfer mode).[1] A format that can accommodate voice, data, and image (television) information simultaneously will be the encoding technique in the future. Currently, ATM has this capability; however, Internet protocol (IP) will be favored if Internet connections can be made with greater reliability.

For the 2 billion subscribers worldwide (as of the year 2000), bandwidth demand for telephone, Internet connections, and high-definition television (HDTV) will continue to increase exponentially if cost can be decreased by integrating devices and by transmitting along one medium—optical.

Wireless

The wireless market has exploded. Extremely high growth rates of 67% in 1999 and 47% in 2000 have demonstrated the demand from consumers. However, available bandwidth is quickly being utilized. The total frequency range available for wireless is 1 Hz to 100 GHz, thus providing a maximum bandwidth of 100 GHz. Only a fraction of this, however, is available for public communication with the remainder used exclusively by the government for military and scientific purposes. In addition, much of this spectrum is attenuated by water vapor or requires line-of-sight operation. The common bands for wireless telecommunication are the following in terms of center frequency and bandwidth in parentheses: 800 MHz cellular (25 MHz bandwidth), 1.9 GHz digital personal communication system (PCS; 200 MHz), 50 GHz satellite (500 MHz and 2.5 GHz).[1] Due to limited wireless bandwidth, data transfer with currently available portable devices is very slow. The so-called 2G (second-generation) wireless is rapidly becoming available across the United States and is already implemented throughout Europe and Japan. Typical data transfer rates are less than a 56K modem. Due to the challenges presented in 3G (third-generation) technology,[8] an intermediate solution, 2.5G, will be necessary to satisfy market demand for lower cost networks and handsets. Although bandwidth is limited, wireless has evolved greatly from the first-generation analog to digital (2G) service, which uses digital signal processing to reduce static and cancel white noise in addition to optimizing access.

The method used to divide the wireless spectrum for individual users is known as access. Three types of access have been developed: frequency division multiple access (FDMA), time division multiple access (TDMA), and code division multiple access (CDMA). Current wireless networks utilize TDMA and CDMA due to the efficient use of bandwidth (i.e., more phones can access the same bandwidth). The promise of 3G technology is likely to enable a society with the world at their fingertips. With fast data transfer rates, the internet will be instantly accessible. In 20 years from now, telecommunications will have evolved into a seamless communication network accessed by wireless technology.

1.2 Transmission

Voice

The current telephone network provides highly reliable dedicated connections between telephones based on a circuit-switched system. This is obvious in the case of wired connections where each phone call is connected when the handset is answered. For wireless communication, both analog and digital generations dedicate bandwidth for each individual user. In a wireless system given an allotted bandwidth segment, part is used for transmitting to the mobile user and part is used for receiving, thereby providing a dedicated connection.

Dedicated line connections yield the quality expected in a telephone network, for example, minimal delays, unnoticeable echoes, and high clarity.

Data

The Internet has spawned a relatively new form of transmitting information via broadband multimedia networking. Data packets are sent over the network using Internet protocol (IP). This allows for multiple routing and error correction routines that differ dramatically from voice networks. Foremost, IP allows for very inexpensive networking. However, in terms of telecommunications, packet delays makes a conversation between people extremely frustrating. Just in the same manner as delays from live satellite interviews seem like brief eternities. Typical IP connections produce longer and unpredictable delays, which is the main reason that the Internet is not currently used to carry voice traffic. When a more reliable connection can be made using IP, then clearly it will be preferred for the transfer of voice, data, and video.

The details of the transmission of information over a communication network are not of obvious importance to the materials scientist or hardware engineer. However, the designer of future systems must be aware of the limitations of current networks and access protocols in order to anticipate the evolution of communication hardware. Current materials and devices are outlined in the following section.

2 MATERIALS SELECTION FOR SELECT COMPONENTS

Selecting a material for a particular component is an extremely difficult task. In an ideal world, the person designing a particular network realizes that the effective frequency range is, for instance, 1 GHz. In their design, black boxes represent transmitters and receivers. However, no details are presented on how to fabricate such a device. Therefore, the electrical engineer is asked to design a receiver for a 1-GHz device with 10 MHz bandwidth. The picture thus drawn includes filters, amplifiers, capacitors, resistors, and the like. The filter performance requirements, however, surpass any component on the market. Therefore, the engineer speaks to a physicist who determines that the only way to make a large bandwidth filter is to utilize a piezoelectric material with a sufficiently large electromechanical coupling coefficient. Finally, the physicist speaks to a materials scientist who lists the materials with such coefficients or embarks on a search for a new material with such characteristics. The materials scientist also must select the best method to produce (or synthesize) such a material in the appropriate geometry for the component, such as, single-crystal, polycrystalline monolith, epitaxial thin-film, amorphous thin-film, or polymer coating.

This design chain is not the end, though. The above picture is not complete for devices, components, and networks in the real world, which must satisfy a long list of technical and cost requirements. For instance, an electro-optic material, which demonstrates a change in index of refraction (n) with applied electric field (E), could be used for a number of applications if it meets the requirements given in Table 1. As a materials scientist, tuning or optimizing one variable is challenge enough. However, to meet a list such as given in Table 1, thousands of choices are available, yet none may be feasible. For example, most electronic and optical devices utilize either pure elements, alloys, or simple binary compounds (two elements only, e.g., GaAs). The semiconductor laser materials, however, incorporate three or four elements in order to tune the bandgap. This compositional variation occurs in a pseudo-binary structure. Even though many elements are present, there are only two unique sites in the atomic structure for the atoms to reside. The structures for silicon (Si) and indium phosphide (InP) are illustrated in Fig. 4. Immediately, many similarities are observed such as the following: cubic symmetry, equal numbers of atoms in the unit cell, and tetrahedral coordination. However, only one atomic site is unique in the structure of Si, whereas two atomic sites are present in InP (A site and B site). Both sites are tetrahedrally coordinated, but the (0, 0, 0) position at the corner of the unit cell is fully occupied by P and the symmetry position at ($\frac{1}{4}$, $\frac{1}{4}$, $\frac{1}{4}$) is fully occupied by In. In the case of a gallium indium arsenide phosphide [(Ga, In)(As, P)], the elements are substituted in these positions as a random statistical population of each chemically equivalent element on a given atomic site. To elucidate, Ga and In are chemically similar in that both have +3 oxidation state with similar valence shells; and therefore, both reside on the ($\frac{1}{4}$, $\frac{1}{4}$, $\frac{1}{4}$) site. In the same manner, As and P are similar possessing −3 oxidation state and residing on the (0, 0, 0) site.

Due to this discrepancy between tailored properties and ease of manufacture, many materials currently in devices is very limited. Whereas, the number of

Table 1 Properties of Currently Used Electro-Optical Material, Lithium Niobate ($LiNbO_3$), and Desired Properties for New Electro-Optical Material Incorporated into Silicon Optical Bench Applications (SiOB)

Properties	$LiNbO_3$	New Material Requirements
Linear electro-optical coefficient	$r_{33} = 31$ pm/V	$r > 2$ pm/V
Optical attenuation (at 1.55 μm)	$\alpha < 1$ dB/cm	$\alpha < 10$ dB/cm
RF attenuation (10–100 GHz)	Low	related to α
DC resistivity	$\rho \sim 10^{14}$ Ω·cm	$\rho > 10^{6}$ Ω·cm
Index of refraction (at 1.55 μm)	$n = 2.17$	$1.2 < n < 2.5$ (lower is better)
Dielectric constant (at RF)	$\varepsilon = 29$ (along a) 44 (along c)	$\varepsilon = 10$–100 (lower is better)
Waveguide contrast (Δn) maximum	0.5% for Ti-diffused	1%
Materials compatibility with SiOB	No	Desired
Postprocessing temperature	500°C	<1100°C for SiOB
Thermal stability	$T_c = 1165$°C (Curie temp.)	$-60 < T < 100$°C
Breakdown field	$E > 10$ MV/m	$E >$ operating field (~1 MV/m)
Depoling field	$E > 8$ MV/m at $T = 130$°C	$E >$ operating field (~1 MV/m)
Creep or fatigue	No	Typical for most Pb-based ferroelectrics

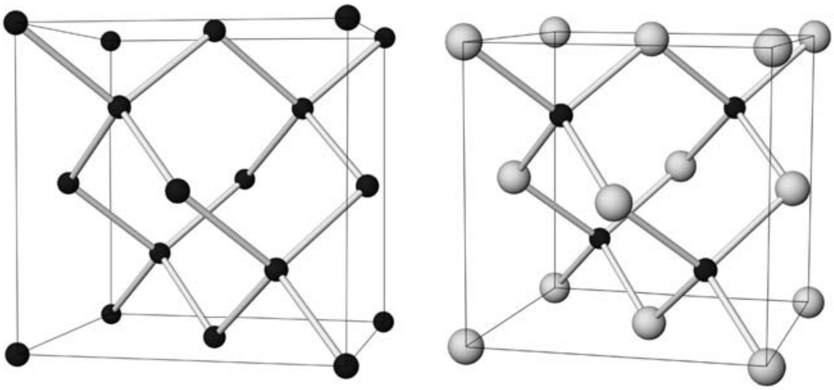

Fig. 4 Unit cells of the Si (left) and InP (right, In - dark, P - light) structures.

materials discussed in the scientific literature for application to telecommunications is overwhelming. Only innovation will bring some of these exciting materials into commercial use.

2.1 Packaging

A package can have many different functions: providing protection, distributing power and signal, and dissipating heat.[9] The ability of a package to perform these functions is dependent on the device as well as the materials properties and design of the package. For example, devices with robust passivation and protective layers may require a less robust package. Integrated circuits and devices relevant to telecommunications contain layers of conducting, insulating, and passivating layers, each with different criteria regarding protection from the environment.

The telecommunications industry continues to grow, and its growth is enabled by the ability to package its devices. Plastic packaging will play a major role in its future. Oftentimes, however, packaging is a secondary consideration in the development of silicon-based devices. Consequently, packaging has received far less attention than warranted, given its crucial role in reliability, performance, and cost. The tide is shifting as device fabrication continues to explore ways to reduce cost and improve performance.

In order for optically interconnected systems to be manufactured realistically, the packaging issue must be addressed for some telecommunication devices, reliable packages have electrical and optical interfaces and must also be able to withstand shock, vibration, and temperature variations. Ideally, these packages are also of low cost.

Packaging is an area that is common to all forms of telecommunications: cable, optical, and wireless.

Packaging Materials

A host of packaging materials is used in integrated circuits and thus telecommunications. They include metals, ceramics, plastics, and glasses. Packages that are impervious to the environment (hermetic packages) are usually made from metal, ceramic, or glass materials; plastics make up nonhermetic packages. In this section, we will survey packaging materials and their uses. Sample applications will also be discussed.

Ceramic Packages. Ceramic packages are laminate sheets of ceramic powder made from a slurry of ceramic powder and a liquid binding mixture of polymers and solvents. Sheets are cut into the appropriate size and via holes are punched. These via holes are filed with metal powder and conducting traces of metal powder are printed on the surface to form circuit patterns. These sheets (as many as 30) are aligned and laminated. Additional circuit lines are metallized, and the leads to them are attached. As a whole, ceramic packages have several benefits, which include hermaticity and the ability to sustain multiple input/output ports. Alumina-based ceramic packages are ubiquitous in optoelectronic devices due to superior radio frequency (RF) characteristics and high reliability.[10] However, these packages have relatively high cost, poor dimensional precision on firing, and a high dielectric constant.[11]

Refractory Glass Packages. A lower cost alternative to ceramic is refractory glass. Although glass has less complexity than laminate ceramic sheets, it is a reliable and hermetic option. Typical glasses used are $PbO–ZnO–B_2O_3$ types with melting points near 400°C.[11] Although hermetic, glasses can at times trap moisture and impurities within the package and thus suffer imperfect reliability. The combination of glass with lead frames made of Kovar (Fe–Ni alloy) has been found to work well and makes up the technology known as CERDIP, which denotes the used of ceramics (CER) in dual-in line packages (DIP). CERDIP packages may use solder glasses which, specifically, are not glasses, but ceramics.

Metals. Solders and their alloys[12] are commonly used for die attachment. They include: tin,[13] tin–lead,[14] indium,[15] and gold eutectic alloys (with tin, silver, or germanium).[16] In one approach, stable bonds of gold eutectic alloy solders have been made with multilayers of alternating gold and tin.[17]

Metals are also employed as diffusion barriers in laser die attachments to reduce the unwanted development of intermetallic compounds.[17] Tungsten can reduce the diffusion of gold from the die into indium solder. Nickel prevents copper from diffusing into indium as well. To promote reliable bonds, wettable layers are also used. Nickel–tin improves wetting of barrier layers to gold–tin alloys. In addition, metals and semiconductor materials are used as heat sinks in optoelectronic die attachment[17] and include materials such as copper, copper–tungsten, silicon and silicon carbide, silver, and variations of each of these. Metals (Al, Cu, Au, and their alloys) are also used for interconnection.

Plastics. The number of types and uses of polymers in packaging is formidable. There are excellent discussions of them in the literature.[9] Plastics may be used as dielectric layers or adhesives and in constructing the body of the moldings. In this section, we will explore them according to their function.

1. Large-scale integration requires insulating layers between metals. High-temperature polymers serve as dielectrics between such layers and have excellent planarization properties, making them well suited for integra-

tion. Polyimides represent a class of materials used as interlayer insulators. Usually spun-coated, they form conformal coatings with excellent thermal and electrical properties. Their ability to form conformal shapes also renders them useful in creating conformal coating layers as well.

2. Besides polyimides, silicon rubbers serve as conformal coatings. Silicon rubbers are also used as encapsulate materials that are globbed on top of devices and modules for protection. Other polymers that serve this function are Paralenes (poly-*p*-xylyenes), which are applied at low temperature through gas-phase polymerization and provide good electrical and environmental performance.

3. In the manufacture of integrated chips, dies are separated from the wafer and attached to the lead frame with adhesives. Solder bonding alloys and organic adhesives are commonly used. Conductive polymers are a low-cost alternative to gold containing solders and can be used in automated processes. These compounds contain silver flakes that provide an electrical path. Also, the molding package itself is polymer based. Often coated with dielectric coating to provide electrical isolation, moldings are made from epoxy resins, hardeners, fillers, catalysts, and colorant.

Case Studies

Semiconductor Laser and Optical Amplifier Packaging. Packaging has many functions, as noted above, but in the optical arena, it also provides a means of alignment of fiber arrays and/or laser diodes. Various ways of coupling optical fiber to devices, optical alignment, and packaging approaches are discussed in the literature[17] and will not be explored here. It is important to note, however, that much work remains to be done in developing low-cost, precisely aligned packages for integrated optical devices.

A number of thermal, mechanical, optical and electrical factors complicate efforts to package lasers and amplifiers. Among these are the following: lasers have high thermal sensitivity; many devices are sensitive to stress; device performance is often dependent on the ability to suppress Fabry-Perot effects, and parasitic electrical behavior of packages impairs device response.[17]

For example, the connection of optical fibers to waveguide devices requires precise fiber alignment, with lithographically etched fiber guides. Fibers are usually butted against the waveguide materials that lie adjacent to the active element. These elements are held in place with solder bumps that provide alignment and electrical connection. As describe above, many materials interplay in these packaging assemblies underscoring the importance of selecting and designing them.

MEMS Packaging. The general sense of microelectromechanical systems (MEMS) technologists is that MEMS packaging presents the greatest challenges faced in the industry. To further complicate matters, packaging issues are application specific and may not translate well from one device to another. As a result, a few representative cases including telecommunications will be discussed.

Accelerometers are currently used in airbag deployment devices, but future applications are on the horizon. In this device, long cantilever beams support a massive beam. Along the beam are elements of a capacitor that sits between two fixed plates. Motion is detected when the spacing between these elements changes. As one can image, in the case of an airbag, the performance of these devices is vital. There should be no impediment to sensing deceleration. As a result, hermaticity is imperative and has been achieved successfully with CER-DIP hermetic packages.[18] Analog devices have developed a successful scheme to precisely orient the package and maintain the sensitivity of the device.

Ink-jet technology, dubbed the first MEMS technology, requires a different set of criteria. In this configuration, a heater element forms microscopic bubbles that eject ink through a silicon-based nozzle. Here, a package must not block the jets of ink, yet it must be protected from them. One approach is the creation of a flexible circuit array bonded to the ink jets with a polymer.

Optical MEMS products add another layer of complexity, as access to the outside environment is required. In these products a light path is commonly needed to provide light to the "eyes" of the MEMS device. Although the requirement seems simple, it is a complex task to achieve. As a result of these rigid constraints, most optical MEMS systems require vacuum hermaticity to ensure proper operation of the sensitive components.

Although MEMS packages are application specific, there are some commonalities among them. For one thing, the device cannot be obstructed and must be covered by a protective cap. Here, packaging occurs on the wafer level. Another method of wafer level packaging is a method called "flip-chip." It entails the use of a silicon cap with circuitry bonded on top with an encapsulant. The sealing is done by metallization (diffusion solder) or epoxy. An underfill is used to fill the gap between the chips.

2.2 Solid-State Semiconductor Lasers

Advances in material properties have led to ubiquitous deployment of solid-state lasers in CD players, barcode scanners, and optical communications. Specifically, lasers with output centered around 1.3 or 1.55 μm have been prepared using molecular beam epitaxy (MBE) and metal–organic chemical vapor deposition (MOCVD) to deposit thin films of $(In_{1-x}Ga_x)(As_{1-y}P_y)$ solid solutions, hereafter referred to as InGaAsP.[19] The wavelength of the laser light can be selected by choosing a desired lattice constant and then locating the proper composition (Fig. 5). This allows the materials scientist to lattice match to a particular substrate and tune to the operating wavelength, which is related to the bandgap energy of the semiconductor. InGaAsP has been selected as the ideal material for telecommunication lasers because of the following properties: (a) a small direct bandgap that allows for efficient conversion of electrical energy to light at the desired wavelengths, namely 1.28–1.62 μm, (b) substrates of InP grown by the Czochralski technique are commercially available, and (c) technology to form waveguides has been developed.

The microelectronics industry has migrated toward silicon-based devices, even though GaAs demonstrates a higher electron mobility. However, neither of these candidates possess the qualities of InGaAsP. Specifically, silicon possesses an indirect bandgap that prohibits the radiative recombination of electron-hole pairs. Further, GaAs with superior electrical performance and a direct bandgap emits light in the visible spectrum when doped with aluminum. For this reason, (Al,Ga)As devices are found everywhere as red light-emitting diodes and lasers;

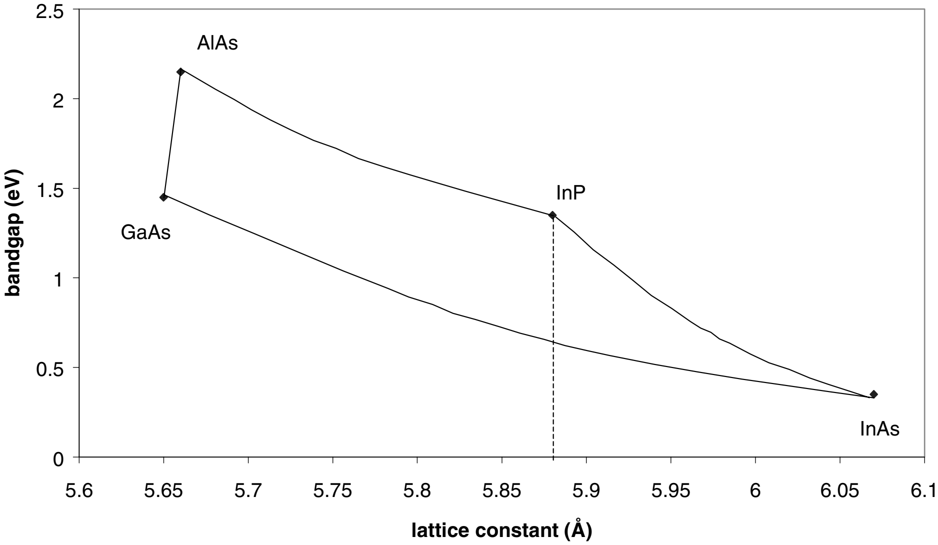

Fig. 5 Bandgap and lattice parameter as a function of composition for $(In_{1-x}Ga_x)(As_{1-y}P_y)$ solid solutions.

however, the bandgap energies required for telecommunication are much lower. Therefore, all of the processing steps that were developed for Si and GaAs devices were duplicated for InP chemistry.

State-of-the-art telecommunications systems use 80 wavelengths separated by 50-GHz spacings. Control of the MOCVD growth conditions for solid-state lasers is paramount for a reliable process where layer thicknesses can deviate by only a few angstroms. The typical InGaAsP thin-film stack to fabricate a laser cavity are illustrated in Fig. 6. Transmission electron microscopy (TEM) of an InP semiconductor laser clearly shows layer thickness and grain structure and also indicates quality of epitaxial films as seen in Ref. 20.

Many functionalities have been incorporated into solid-state lasers, which has been reviewed in detail for telecommunication applications.[21] The materials used in these laser devices are all similar, yet the device designs are radically different. The goal is to integrate as many functionalities as possible while keeping reliability high and cost low. For example, distributed feedback (DFB) lasers can produce continuous-wave light at a very specific wavelength, which is required for DWDM since the channel spacing is only 50 GHz. Further, due to the chromatic dispersion of optical fiber, the spectral width of the laser emission is minimized to avoid broadening of the light pulse during propagation in the fiber. A narrow spectral width is accomplished by fabricating a Bragg grating beneath the laser cavity (Fig. 7). The laser cavity is a multilayer quantum well (MQW) structure that provides higher gain, a lower threshold, and lower losses.

Electroabsorption modulated lasers (EML) are an integrated laser and modulator on an InP substrate. Current devices can operate at 10 GHz, and research is pushing the modulation rate to 40 GHz. Further integration is achieved in wavelength-selectable lasers with gain, and modulated intensity are being de-

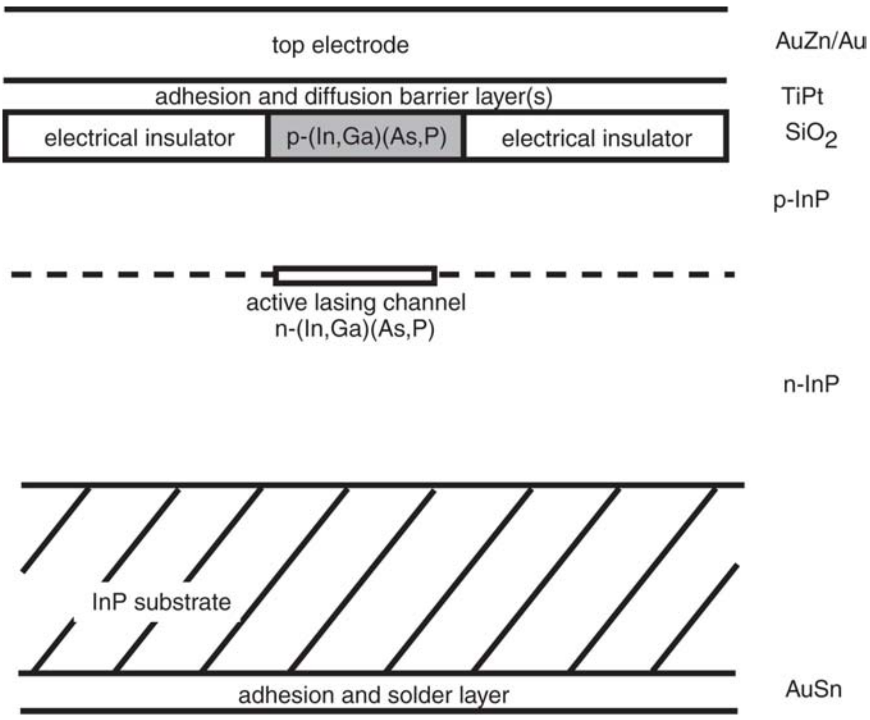

Fig. 6 Semiconductor laser cross section with emitted light from the buried channel active region.

veloped (Fig. 8). For this device, each DFB laser produces continuous-wave light at a specific wavelength. The system selects which laser to activate; then that light passes through the combiner and into a semiconductor optical amplifier (SOA), which boosts the intensity, after which the light is modulated by the electronic data stream. High-performance laser packages, which monitor output

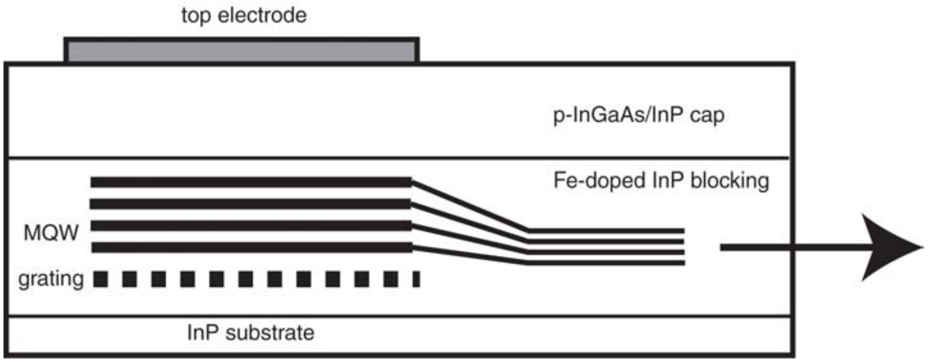

Fig. 7 Cross section of a distributed feedback (DFB) laser.

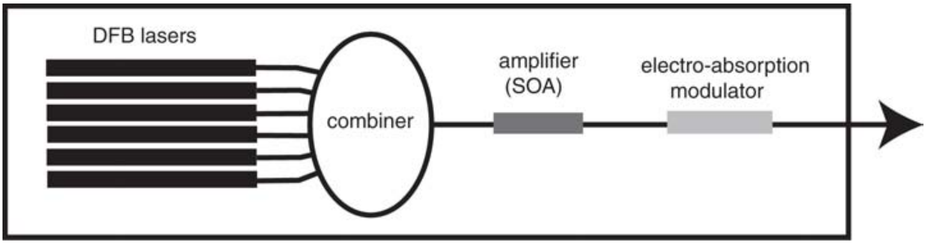

Fig. 8 Schematic top view representation of a wavelength selectable laser.

from the laser cavity, can actively control the wavelength of the device by changing the temperature of the InGaAsP laser chip. The typical materials used in a solid-state laser package are illustrated in Fig. 9. The major assemblies include an InP substrate with InGaAsP laser, a thermoelectric cooler with feedback loop to control temperature, an isolator made by a Faraday rotator and polarizers (see Section 2.8), spinel and glass lenses, an etalon to monitor the wavelength of the laser output, and a photodetector to monitor light output from the back facet, and heat sink subassemblies. The subassemblies are attached by soldering and wire bonding. The assembly is mounted in an alumina-based hermetic ceramic package. Finally, the fiber is aligned and the steel ferrule is laser welded. The future challenges for higher performance solid-state laser devices are immense.[22]

2.3 Photodetectors

Two main types of photodetectors are used in telecommunications: pin (*p*-intrinsic-*n*) and APD (avalance photodiodes).[23] APDs are a high-performance ultrafast photodetector that provide gain for weak signals. APD devices are expensive; therefore, if use of pin photodiodes is possible, it is the preferred choice. In addition, APD detectors require large biases on the order of 50 V, whereas pin devices usually are biased about 5 V. Again as in solid-state semiconductor lasers, the materials are chosen based on bandgap and ultimately on quantum efficiency. For telecommunications, the chosen materials are based on solid so-

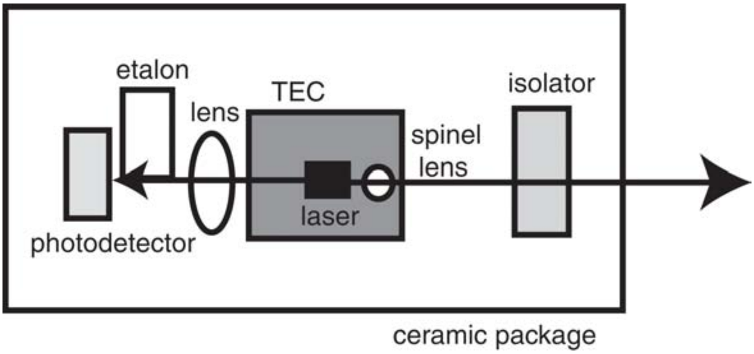

Fig. 9 Basic schematic of a high performance laser package.

lutions of InGaAsP on InP. To improve device performance, a lattice match with the substrate is desired in order to minimize the dark current and thus improves the signal-to-noise ratio of the device.

Additional improvements can be made by appropriate metal contacts. The choice for metal contacts on semiconductors can be an art. For example, AuGe/Ni has been used on InP and TiAu on In(As, P).[24] The selection of a metal contact is initially made depending on the doping state of the semiconductor. The work function (ϕ) of the metal needs to be less than the work function for n-type and greater than the work function for p-type semiconductors in order to make an ohmic contact. The work function is defined as the difference between the Fermi energy and the ionization energy.

2.4 Optical Fiber

The principle of guiding light within a medium is not new. In 1854, John Tyndall demonstrated that a stream of water could guide light.[25] In his experiment, a light shines through a stream of water jetting out from the side of a tank of water would and bends along the stream's path (see Fig. 10). The stream of water successfully guides light until the turbulence of the stream's surface made the beam more diffuse. The water guides the light by internally reflecting it. That is, the light at the water/air boundary is reflected back into the water. Internal reflection occurs only when going from a material of high refractive index (water) to a material of lower refractive index (air).[26]

Today we have more sophisticated means of guiding light, one of which is through the use of optical fibers. Optical fibers are narrow strands of glass, polymers, or a composite of both. One of the early identified challenges of guiding light through a single medium, such as glass, is that light leaks whenever the medium contacts materials other than air, that is, material with a refractive index greater than air. The solution to this problem was addressed by making a fiber "sandwich," placing an outer cladding layer around the fiber core. In this manner, light travels along the fiber core confined by the lower refractive outer layer. This arrangement not only confines the light but also prevents leaks should the fiber touch materials that are more highly refractive (see Fig. 11).

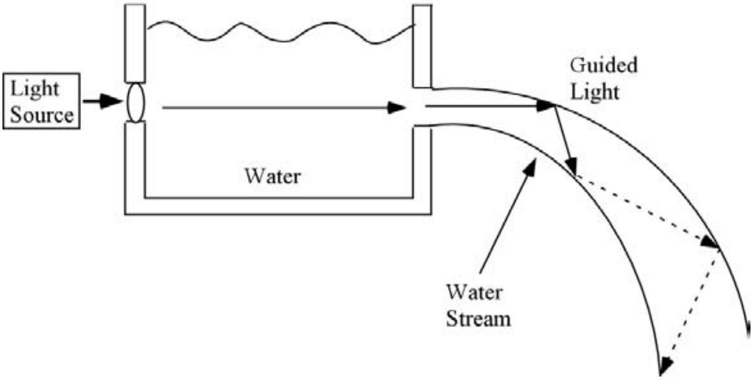

Fig. 10 Light guided down a water stream demonstrating principle of internal reflection of light.

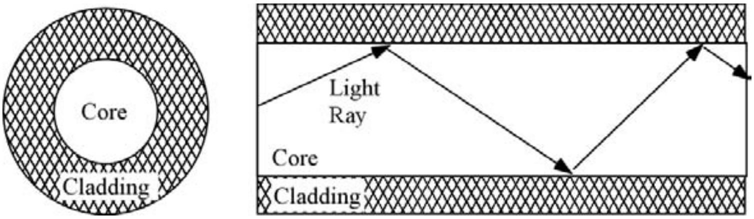

Fig. 11 Light guided in an optical fiber by total reflection in the high refractive core bounce back by the low refractive cladding.

The majority of fibers are made from glass consisting of silica (SiO_2) or silicate. Glass fiber properties have a range that suits both short- and long-haul transmission. Plastic fibers are used less widely and are limited to short distances but can withstand more abusive environments due to greater mechanical toughness compared with glass. In the design of optical fibers it is important to consider the loss or reduction of signal as it travels in the material. Sources of loss in silica fibers are absorption from impurities, absorption due to molecular vibrations, and scattering by fiber atoms.[27] These losses can be resolved by reducing the number of impurities, as well as by optimizing the material's dispersion and tailoring the chemical composition to create graded refractive indexes. For the silica fiber, the chromatic dispersion is near zero at 1.33 μm and has its lowest attenuation at 1.55 μm.[28]

In the next following sections we will examine the materials used in optical fiber and their fabrication.

Glass Materials

Glass optical fibers are nearly always vitreous silica doped with small amounts of germanium oxide (GeO_2), phosphorus oxide (P_2O_5), or fluorine, but can be mixtures of oxides, sulfides, and selenides[29] that create a random covalent network. Unlike ordered crystalline materials, there is no well-defined pattern and accordingly no well-defined melting point.[30] The largest class of optical fibers is made from oxide glasses, in particular silica (SiO_2), with a refractive index of 1.458 at 850 nm.[31] The refractive index of a material may be changed by controllably adding impurities or dopants. Such dopants include fluorine and metal oxides. Inclusion of GeO_2 or P_2O_5 increases the index, whereas the inclusion of fluorine or B_2O_3 decreases the refractive index.[31]

Silica's properties make it a formidable candidate for optical fiber due to its low thermal expansion, high transparency, and high resistance to thermal shock. However, the high melting point needed for its fabrication from the molten form gives rise to the need to explore alternative low-melting-point silicates. These materials include soda-lime silicates, borosilicates, and germanosilicates. Soda-lime silicate glasses are combinations of silica, alkaline oxides (such as Na_2O or LiO_2) and other oxides (MgO, ZnO, or BaO).[31] As a result, these materials are easy to melt and fabricate into fiber.

In some cases it is necessary to boost the signal periodically due to attenuation and losses. The signal can be amplified by doping the fiber with erbium. The EDFA (erbium-doped fiber amplifier) has proven to be an enabling technology for optical communication.[32] A pump laser at 0.98 or 1.48 μm excites the erbium atoms, which transfer their energy an additional 1.55 μm light waves by laser action to amplify the weakened signal.[27] The f-orbital transition in Er^{3+} yields the amplification around the 1.5-μm band used for long-haul telecommunication. The promise of a low-cost alternative to the EDFA is the EDWA (erbium-doped waveguide amplifier). An advantage of EDWAs is the ability to be integrated with other optical components due to the fabrication of the waveguide on a silicon optical bench.

To increase the channels, it is necessary to extend the bandwidth beyond 1.5 μm. Presently, a combination approach of rare-earth doped fiber amplification and Raman amplification is employed.[27]

Another variation of the structure of optical fiber is the development of periodically varying index in the fiber core, called a fiber grating.[33] By selectively transmitting wavelengths and reflecting others depending on the pattern spacing, these fibers can be modified to act as mirrors, sensors, filters, or resonant cavities.[34]

Plastic-Clad Fibers

For short data transmission ranges (several hundred meters) where high losses are tolerable, plastic-clad fibers are an inexpensive alternative. The fibers are composed of silica cores with lower refracting plastic cladding made of silicone resin (with an index of 1.405 at 850 nm) or perfluoronated ethylene propylene (Teflon) (with a refractive index of 1.338). Only used for step-indexed fibers, the core diameters are larger (150–600 μm) than conventional all-glass cores and cladding systems and permit low-cost interfaces with light sources and detectors.

Plastic Fiber

For short distances (maximum 100 m) an all-plastic multimode step-index alternative may be used.[35] Although they have significant signal losses, they are tough and durable and thus do not require special handling. High refractive differences between core and cladding make this an attractive alternative for short haul applications, but costs are still high. Core and clad combination of a polystyrene (index 1.6) core and methyl methacrylate (index 1.49) cladding have been demonstrated.[31]

Fabrication of Optical Fibers

All-glass optical fibers are made of extremely pure silica glass. The cladding is made of the purest material, while the core material includes deliberately and precisely controlled amounts of impurities (which increase its refractive index). Two common methods are used in the manufacture of all-glass optical fibers[34]: They are direct-melt processes and vapor-phase oxidation processes.

In the direct-melt method, the optical fiber is made directly from molten silicate glass. Two concentric platinum crucibles, one for the core and one for the cladding, are filled with the appropriate molten glass. A fiber is then drawn from the orifices at the bottom of the crucibles, rendering a continuous drawing process.[31,34] Despite the relative ease of this manufacturing technique, careful attention must be paid to eliminate contamination of the crucible and furnace environment.

In the vapor-phase oxidation process, highly pure vapors of metal halides (e.g., $SiCl_4$ and $GeCl_4$) react with oxygen to form a white powder of SiO_2 particles. These particles are collected and are sintered by one of a variety of techniques to form a clear glass rod or tube. This rod or tube is called a *preform,* which is as much as 100 mm in diameter and 100 cm long, yielding hundreds of kilometers of fiber. Fibers are drawn from preforms in a precision draw tower furnace. In it, the bottom is softened so that a thin filament of glass (optical fiber) can be drawn. The thickness of the fiber is determined by the drawing speed.

The first optical fiber was manufactured by outside vapor-phase oxidation (OVPO) at Corning Works.[36] A layer of silica particles called soot is deposited from a burner onto a bait rod, where a porous perform is made layer by layer (as depicted in Fig. 12). When the desired thickness is attained, the bait rod is removed, and the porous perform is then vitrified and collapsed into a clear glass rod in a dry and hot environment (above 1400°C). Other methods exist and are described in other texts.[31,34] One method, which is a new fabrication point of view, known as sol–gel, starts from a liquid precursor [e.g. $Si(OC_2H_5)_4$ hydrolyzed in water], is dried and sintered into a glass perform.[34]

One important type of vapor-phase fabrication is the modified chemical vapor deposition (MCVD) created at Bell Labs.[37] The process starts with a very pure tube of silica glass (SiO_2), about 15 mm in diameter and 1 m long. Reactant gases of metal halide gases and oxygen flow through a revolving tube, as depicted in Fig. 13. As the SiO_2 particles are deposited, they are sintered into a clear glass by a moving oxyhydrogen torch, which travels back and forth along the tube. Successive layers of SiO_2 followed by GeO_2 doped SiO_2 are deposited. When the total deposit reaches the desired thickness, the vapor flow is stopped, and higher heat is applied to collapse the solid tube. Glass fibers are surprisingly strong. The longitudinal tensile strengths of 5 GPa in a short length of glass fiber is larger than the yield strength of steel wire (2 GPa). Unlike steel, glass does not yield and does not deform plastically. Glass fibers can elongate 1% more than 10% before fracture occurs. These extraordinary mechanical proper-

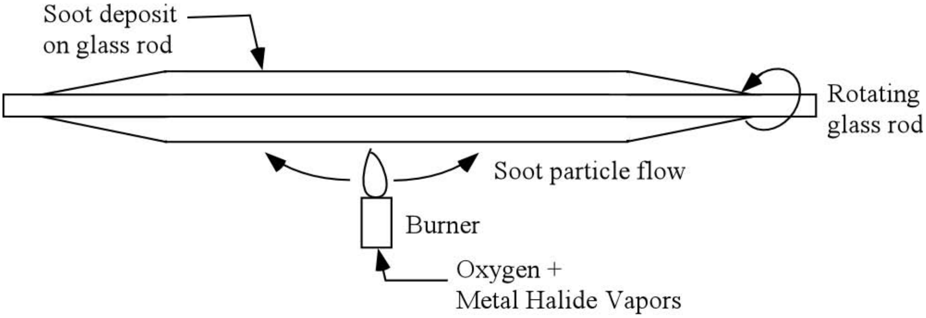

Fig. 12 Soot deposition onto a rotating rod by the outside vapor deposition (OVD) process.

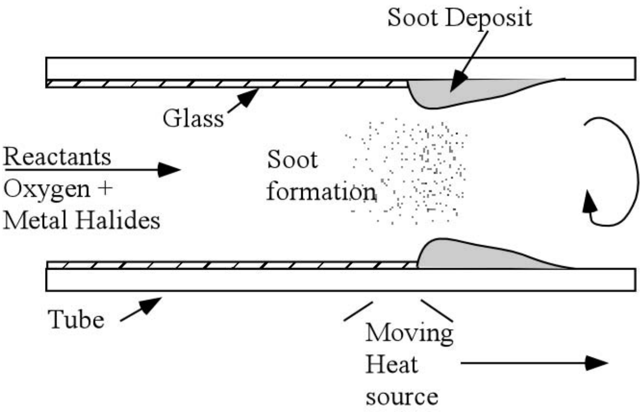

Fig. 13 A schematic of the modified chemical vapor deposition (MCVD) process.

ties of glass fiber derive from the pristine (flaw free) surface of a freshly drawn fiber. In the drawing process, the fiber is quickly coated with a polymer layer to protect the surface from mechanical scratches and chemical reactions with the atmosphere. Commercial optical fibers are routinely proof tested at levels of 0.69 GPa or 1.38 GPa before sale.

Fiber-Optic Cables

Special cable designs are required because of the mechanical properties of glass. These designs are greatly dependent on how the cable will be used. For example, cable may be pulled underground, submerged underwater, or installed outdoors. Generally, a fiber is coated with buffer material and placed loosely in a polyethylene tube. The tube is surrounded by aramid yarn to provide strength and encapsulation. Finally, an outer jacket of polyurethane, polyethylene, or nylon binds the encapsulated fibers together. Larger cables are fashioned from these units by binding fiber bundles to a support with tape, then surrounding them with an outer jacket. Undersea optical fiber cable, like copper cable, needs to be protected from water vapor and its mechanical strength needs to be increased. This is accomplished by forming cables with steel wire strands and polymer jackets (see Fig. 14).

Fiber Devices

Advances in optical fiber have extended into device fabrication, such as fiber Bragg gratings (FBG).[38] FBGs are made by doping the core of an optical fiber with germanium, which increases the index of refraction allowing the guiding of light. This fiber is masked according to the desired modulation pattern and exposed to ultraviolet light, which increases the index of refraction in the areas exposed due to the photorefractive effect. Thus, the core of fiber posseses an index modulation along the propagation direction, thereby demonstrating Bragg grating characteristics. These gratings can be used as simple filters where the reflected wavelength is removed from the transmitted signal. However, many functionalities can be added to the FBG to monitor network operation and to permit rapid reconfiguration. For instance, thin-film heaters can be deposited on

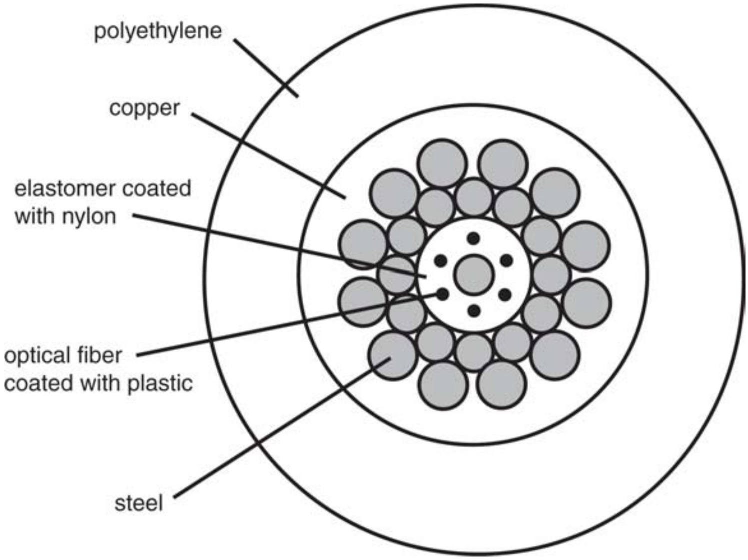

Fig. 14 Cross section of optical fiber cable for undersea deployment.

the fiber, which dynamically compensate for chromatic dispersion during propagation of light in long-haul fiber by adjusting the chirp of the FBG.[39]

2.5 Electro-Optical Materials

The area of optoelectronics is demonstrating exploding growth. In the past few decades, optical communication used fiber as the transmission medium but relied on electronics for amplification, signal processing, and switching. The conversion of information from optical to electronic to optical represents a bottleneck in the optical network. It is obvious that faster data transfer will only be possible if the electronic components are replaced by all-optical counterparts. To manipulate light, materials demonstrating nonlinear optical (NLO) phenomena are necessary. Materials with large electro-optical coefficients also demonstrate significant NLO behavior. One requirement for all electro-optical materials is a lack of inversion symmetry in the crystallographic space group, also known as acentric.

Piezoelectric materials are acentric with a crystallographically defined polarization direction. This direction must be oriented (textured) as deposited if in thin-film form. For nonferroelectric materials, such as ZnO and AlN, poling is not required and fatigue is not observed. However, usually nonferroelectric materials have low electro-optical coefficients and only demonstrate a linear electro-optical coefficient. These materials are not applicable in nonlinear devices that utilize the Kerr effect.[40]

As mentioned earlier, lithium niobate ($LiNbO_3$), is an electro-optical material that demonstrates a change in index of refraction (n) with applied electric field (E). High-speed modulators with rates of 40 Gb/s are commercially available. Waveguides are defined by Ti diffusion, and Au is used as an electrode material. $LiNbO_3$ is a ferroelectric material with a moderate electro-optical coefficient, and it does not display fatigue. However, due to the photorefractive effect, $LiNbO_3$ has a low optical damage threshold limiting it from nonlinear applications that require high-intensity light.

Another ferroelectric, $BaTiO_3$, demonstrates a large electro-optical coefficient ($r_{33} = 100$ pm/V), but the thermal stability is low due to a Curie temperature of 120°C, which is too close to ambient temperature. The Pb-based ferroelectrics (PT = $PbTiO_3$, PZT = $PbZr_{1-x}Ti_xO_3$, and PLZT = $Pb_{1-3y/2}La_yZr_{1-x}Ti_xO_3$) have very large electro-optical coefficients, but their properties exhibit fatigue (i.e., the electro-optical coefficient decreases with time). In addition, optical attenuation is high since large single crystals have not been grown and epitaxial films cannot be grown to thicknesses required for optical waveguides (~5–10 μm). One reason to investigate the Pb-based ferroelectrics is their high electro-optical coefficients (r_c), which in turn lead to a large change of the index of refraction (Δn) in an applied electric field (E) through the following relationship:

$$\Delta n = -\tfrac{1}{2} n^3 r_c E$$

where r_c is approximately $r_{33} - r_{13}$ for tetragonal symmetry.[41] Reported values for r_c are the following (units of pm/V): $BaTiO_3$ 80, $LiNbO_3$ 18, $Pb_{0.88}La_{0.08}(Zr_{0.40}Ti_{0.60})O_3$100, $Pb_{0.88}La_{0.08}(Zr_{0.65}Ti_{0.35})O_3$ 520.

A new direction pursued to find high-efficiency elecro-optical materials is in the area of organic materials.[42] The design of organic molecules to crystallize in acentric space groups with large electro-optical coefficients has been discussed based on geometrical arguments of rodlike dipolar molecules.[43] In addition, inorganic-organic hybrid nanocomposites are being developed for electro-optical applications.[44]

2.6 Antireflection (AR) Coatings

Attenuation of optical signals through lenses can be significant. For example, pure SiO_2 with an index of refraction (n) of 1.5 will reflect 4% of the light from a single air–glass interface.[45] This attenuation can be reduced to 1% using an antireflection coating of MgF_2. The coating consists of a quarter wave film, meaning the thickness of the film is $\frac{1}{4}$ of the wavelength of light that is desired to be transmitted. The optimal index of refraction for the antireflection coating is $n^{1/2}$. Therefore, SiO_2 would benefit most from a film with n equal to 1.22. Such a low index is difficult to achieve; however, MgF_2 has an index of 1.37 at 1550 nm, making it a good candidate as a coating material. In addition, it is easy to fabricate using electron beam evaporation. AR coatings are used on almost every optical element in a device.

2.7 Optical Filters

Selecting an appropriate bandgap and index of refraction, multilayers of inorganic thin films can be designed for use as optical filters. A variety of applications are available: antireflection coatings, reflective coatings (dielectric mirrors), neutral density filters, beam splitters, short-wave pass filters, long-wave pass filters, bandpass filters (monochromatic), minus filters, and shaping filters.[46]

2.8 Isolators Incorporating Faraday Rotators

An isolator prevents light propagating down a optical fiber to reenter a laser cavity, which disrupts the mechanism of stimulated emission. The isolator combines two polarizers with a Faraday rotator. Light emitted from the laser cavity is sent through a polarizer to achieve plane polarized light. Then, this light passes through a Faraday rotator that exactly rotates the polarized light by 45°, which passes through another polarizer tipped at 45° to allow the light to pass out of the isolator. Any light that propagates down the fiber and is reflected back would undergo an additional 45° rotation at which point the initial polarizer would deflect the 90° out-of-phase light away from the laser cavity, thereby isolating the cavity.

An iron garnet, $(Bi, Eu, Ho)_3(Fe, Ga)_5O_{12}$, has been developed as the material for Faraday rotators for telecommunications at 1.55 μm.[47] This material was selected for its isotropic optical properties, which arise from cubic symmetry. The garnet structure can be described by $C_3A_2D_3X_{12}$, where the cations have a $+3$ oxidation state and reside in dodecahedral (C), octahedral (A), and tetrahedral (D) coordination, and the oxygen anions (X) having a -2 charge reside in tetrahedral coordination. To increase the specific rotation, bismuth was added into the dodecahedral site. However, this also increases the temperature dependence of the specific rotation, an undesirable consequence. The reason for increasing the specific rotation is to reduce the thickness of the device. For instance, yttrium iron garnet, $Y_3Fe_5O_{12}$, would require a thickness of 2.7 mm in order to rotate light 45°, whereas the optimized material is only 0.4 mm thick to achieve similar rotation. Gallium doping is used to reduce the saturation magnetization such that the material can only switch between saturated single-domain states. This permits the use of Faraday rotators in applications were an external magnet are prohibited, since the rotator will remain in a single-domain state in the absence of an external magnetic field. In addition, all cations must retain the $+3$ oxidation state to maintain minimal optical absorption at 1.55 μm.

The polarizers can be replaced by a highly birefringent material to make a polarization independent device. Rutile, TiO_2, is one possible material for this application. In this manner, both components of the refracted light (ordinary and extraordinary) can propagate through the device without significant attenuation. Future developments would include thin-film fabrication for integrated optics.

2.9 Microelectromechanical Systems

Microelectromechanical systems (MEMS) is a burgeoning technology that integrates many of the lessons of integrated circuit technology into small-scale systems. The thrust for this technology combines the high volumes of production with low cost. In addition to the economic potential, this approach allows unique opportunities in the full integration of devices at very small scales. A device composed of mechanical and electrical elements built on silicon can now sense,[48] actuate,[49] produce power,[50,51] guide light,[52] and maintain chemical reactions.[53] The success of this technology, however, lies in the understanding of the materials issues associated with the design and fabrication of the tiny devices.

Although the materials set borrows heavily from silicon technology, there are a few new players in the MEMS arena. MEMS technology extends the materials set beyond those of microelectronics to include diamondlike carbon,[54,55] silicon carbide,[56] metals and polymers,[57] and piezoelectric materials.[58] Silicon processing has also changed its character to create high-aspect ratio devices. For example, deep reactive ion etch (RIE).[59] Bosch techniques,[60,61] and other techniques are now used to create deep trenches and other unique geometries that were not compatible with etching anisotropies of conventional materials.

A host of fabrication techniques[62] are used, including surface machining,[63] bulk machining,[64] and molding processes (or LIGA).[65] Surface machining builds on top of the silicon substrate, bulk machining relies on etching into the wafer, and molding produces pieces by microfabricated molds. The basis of micromachining is photolithography, which is the means of defining regions on a wafer. Etching (or removal of silicon), doping, and deposition can all be controlled by photolithography and make up the key ingredients to building three-dimensional forms. Surface micromachining is comparable to the process to build VLSI technology. Molding is based on LIGA, an acronym based on the German phrase for *Lithographie*, *Galvanik* (electroplating) and *Abformung* (molding). This technique can create high-aspect ratio photoresist regions by illumination it with high-intensity X-ray radiation (often done with a synchrotron). The resist is exposed to form open regions in which electroplated metal is deposited. The resulting device consists of photoresist and electroplated metals agglomerations. Other techniques on the horizon for fabrication include conventional techniques such as cutting, shaping, and drilling on a small scale.[66,67] Sand blasting, laser ablation,[68] and spark erosion may become viable paths to microfabrication as well.

MEMS Materials Set

The pathways to fabrication described above provide designers a means of developing a host of devices. However, achieving reliable low-cost devices is strongly dependent on the materials used. The materials set for MEMS consists of all the flavors of conventional integrated circuit technology, plus a few new ones. The original materials include single-crystal wafer substrates, polysilicon resistive layers, and aluminum and copper conductive paths. In addition, silicon oxide, silicon nitride, and titanium nitride are used for insulation, passivation, and passivation/protection, respectively.[69] In the MEMS world, these materials now serve additional functions. Silicon, polysilicon, and silicon nitride are mechanical elements, aluminum is used as a reflective material, and silicon oxide is used sacrificially. Other materials are employed as well. Gold is used as a reflective material and as a conductive path. Titanium and chromium are used as adhesion layers, and silicon-on-insulators (SOI) wafers serve as substrate material. In addition, photoresist was once used and removed in processing; now it remains as a structural material in MEMS.[57] For materials in MEMS, everything that was once old is now new.

In MEMS, mechanical elements for displacement-based sensors include rotating parts and torsional flexures. Silicon is an attractive material due to its high stiffness and high strength. Silicon carbide, diamond, and alumina (Al_2O_3) are also good choices for similar applications. For applications in which high mechanical forces and power levels are required, metals are a more obvious choice. Electroplated metals such as copper, nickel, and their alloys are used, as are other metals such as chromium, iron, cobalt, and strengthened versions of each. Silicon carbide offers a high stiffness, hardness, and wear-resistant option in the MEMS material palette, yet in comparison to other materials in the VLSI collection, its development is not as mature. Promising materials for the future include sapphire, amorphous alumina, fused silica, and diamond, which are available in wafer form to create MEMS devices.[69]

Transducer materials used to sense, actuate, or convert mechanical stimuli into electrical responses are also important to note. Actuation can occur electrostatically, magnetically, piezoelectrically, and thermally. Electrostatic and thermal actuation materials are generally made from conventional thin-film metals. More esoteric piezoelectric and magnetic materials are used in the associated form of actuation. The most common piezoelectric materials include lead zirconate titanate (PZT),[70], zinc oxide (ZnO).[66] and aluminum nitride (AlN).[58] These materials are typically deposited by sputtering; however, a special case of sol–gel deposition onto micromachined parts has also been reported.[71] Magnetic materials such as Permalloy[72] and shape memory alloys such as NiTi[73] are means of actuation.

One example of a MEMS-based device for telecommunications is the optical cross connect, developed at Bell Laboratories.[74,75] This device redirects light between fibers by micromachined mirrors (see Fig. 15). The silicon mirrors are supported by gimbals of the same material. Two sets of torsional silicon bars allow the mirror to tilt in two directions. The mirrors are moved by electrostatic actuation and require passivation layers for protection. Electrical signals for ac-

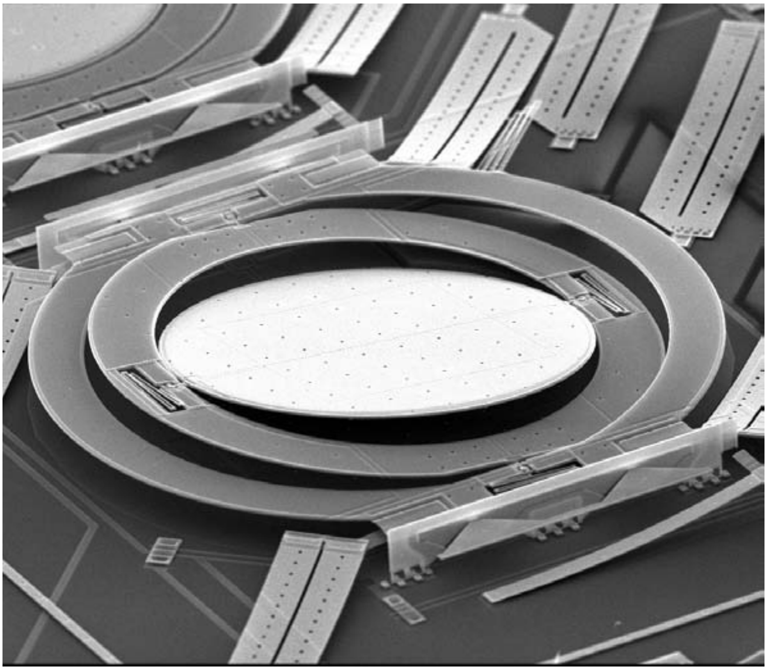

Fig. 15 An optical cross-connect MEMS mirror used to route light from one optical fiber to another.

tuation traverse gold conductive paths. Gold is also used as the reflective layer on the mirrors. This example illuminates the plethora of materials needed to make a device and underscores the need for understanding the materials in order to create reliable devices in this fast-moving technology.

From this discussion we have seen that MEMS devices will certainly be an enabling technology for telecommunications for several decades. Its success is due largely to understanding, creating, and designing of materials. Understanding material properties provide the means to create reliable, low-cost and fast devices. In the light-wave network, a host of possible MEMS applications remain undiscovered. The new capabilities of this technology and the lessons learned from integrated circuit technology are rich fodder for growth in the telecommunication commercial arena.

2.10 Microwave Radio-Frequency (RF) Resonators

A review of materials for microwave applications has been written by Laverghetta highlighting dielectric laminates and metals.[76] The most important properties for microwave dielectrics is the magnitude of the dielectric constant and the dissipation factor.

Bulk Resonators

Most wireless base stations use a bulk dielectric material, $Ba_2Ti_9O_{20}$, for RF resonators. This material uniquely displays a high dielectric constant and low-temperature coefficient of the dielectric constant (TCK). Most dielectric materials demonstrate a nearly linear relationship of the dielectric constant and TCK known as the Harrop correlation (Fig. 16). However, $Ba_2Ti_9O_{20}$ displays an anomalous low TCK allowing for the development of a high-quality factor (Q factor) resonator with temperature insensitivity. Also, $Ba_2Ti_9O_{20}$ is able to withstand the high power requirements of base station transmitters.

Recent advances in high-temperature superconductors have yielded frequency-tunable resonators using copper-oxide-based superconductors for wireless communications. Base station filters and amplifiers are currently made by Superconductor Technologies from thin films of TBCCO (thallium barium calcium copper oxide).

Thin-Film Resonators (TFRs)

TFRs are already emerging in wireless handsets and have several advantages over the conventional bulk dielectric resonator. First, the dielectric resonator is the largest single component is a wireless telephone. In addition, due to the many wireless network standards around the world, a global traveler would need several separate phones. The advent of the TFR brings a device with multiple filters all within an area of a square centimeter and as thin as any integrated circuit. Also, with further development, TFRs will be integrated with electronics on the same wafer leading to reduced cost and even smaller phones.

Optimal materials for these devices are materials having the wurtzite structure, such as ZnO and AlN. To be implemented in 2G and 3G wireless networks, the material must be piezoelectric and have a large electromechanical coupling coefficient that is proportional to the bandwidth of the filter. TFR devices operate

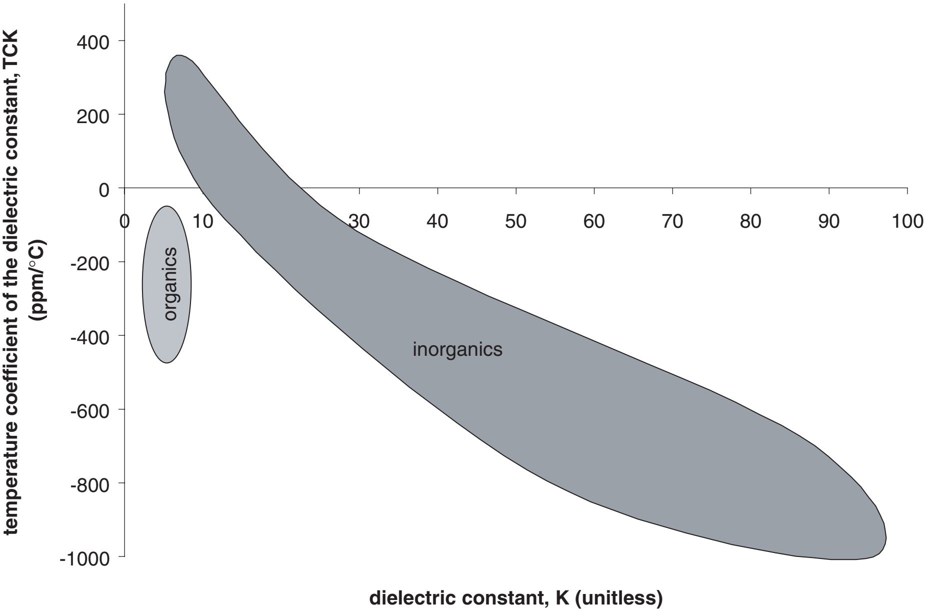

Fig. 16 Harrop correlation for dielectric materials.

based on the propagation of acoustic waves either as a bulk acoustic wave or a surface acoustic wave. Research toward the fabrication of reliable TFRs from ferroelectric materials is underway.

2.11 Repeaters and Switches

Repeaters, also known as regenerators, perform filtering and amplification to electrical or optical signals. For optical communication, it would be advantageous to have an all-optical network including repeaters and switches. For the time being, conventional electronic circuits dominate these systems. Most of these challenges are similar to those experienced in the microelectronics market in order to achieve higher speed: high thermal conductivity materials, such as SiC,[77] high dielectric constants for logic gates and memory capacitors, and low dielectric constant materials that minimize RC delay. These topics will not be covered in this review.

2.12 Multilayered Ceramics

The multilayer ceramic integrated circuit (MCIC) has enabled the production of low-cost RF wireless devices.[78] The devices are fabricated with low-temperature co-fired ceramics (LTCC) to incorporate components such as capacitors, resistors, transmission lines, and inductors into the substrate equivalent of a printed circuit board. The following materials are used in this process: dielectrics (crystalline and glassy phases containing MgO, SiO_2, Al_2O_3, B_2O_3, PbO, and CaO) and conductors (Au, Ag, Cu, Pd/Ag).

Power systems also take advantage of multilayer ceramics for magnetic components with Mn–Zn ferrite cores, such as transformers and inductors.[79] This work led to the innovation of a power supply for a microprocessor nearly the same size as the integrated circuit itself.

Alumina-based multilayer ceramic packages are ubiquitous in optoelectronic devices due to superior RF characteristics and high reliability, as previously described.[10]

3 COMMUNICATION SYSTEM COMPONENTS

Table 2 provides information on various communication system components. The table is not inclusive, and with advances in research and development, the number of new materials in telecommunications is increasing dramatically.

4 SYNTHETIC METHODS

4.1 Introduction

Remarkable progress in the field of telecommunication materials have taken place due to the advances in thin-film processing for deposition and in material fabrication. In this section, a review of such processes needed to create technologically relevant materials will be discussed. Emphasis is placed on offering a working knowledge of each technique and providing a guide to the literature.

4.2 Sputtering

A common technique for deposition is sputtering.[80] It consists of the bombardment of the material of interest, known as the target, with energetic ions. In the sputtering process, an inert gas, such as argon, flows to provide a medium in which the sputtering process can occur. Gas pressures usually range from several to 100 mTorr.[81] Sputtering is induced when a high negative bias is applied to the target, as depicted in Fig. 17. The bias initiates the ionization of the gas, which turns into a plasma consisting of ions, electrons, and neutral species. The positive argon ions accelerate toward the target. When the ions strike the target, they remove target atoms by momentum transfer. The target erodes as atoms or clusters of atoms are removed, condensing on the facing substrate and forming successive layers.

Sputtering is advantageous because the use of energetic sputtered species gives rise to highly adherent dense films of uniform composition and thickness. Commonly used for metallization in telecommunications, sputtering generally preserves the stoichiometry of the source materials. These benefits are often offset, however, by the technique's low deposition rate and the inclusion of sputtering gas.[82]

4.3 Evaporation

The simplest evaporator consists of a resistively heated source. In this configuration, a filament wire, refractory metal sheet, or crucible source is heated. Common materials for the source are tungsten, molybdenum, and tantalum. In all cases, these materials should have a high melting point and low vapor pressure.[83] The source filaments support the materials to be evaporated and during heating are wetted by them before evaporation takes place. Wafers suspended above the assembly are then coated with a thin film of the evaporating material.[84] Although the resistive method of evaporation is easy to set up, it is plagued with contam-

Table 2 Summary of Selected Communication System Components

Material	Synthetic Technique	Property	Component	Function	System/Network
Ag	Sputtering, evaporation, electrochemistry	Electrical and thermal conductor	Optoelectronic packaging	Heatsink (interconnection)	Networks, packaging
Al	Sputtering, evaporation	Conductor, reflective (visible)	Interconnects, optical MEMS	Electrical conductor, reflective surface	Wireless, optical
AlN	Sputtering, CVD	Piezoelectric	MEMS	Sensor/transducer materials, actuation method	Wireless
Au	Evaporation	Excellent optical reflectivity at 1.55 μm	Optical cross connect (OXC)	Mirror used to route optical signals in MEMS OXC	Optical
Au	Sputtering, evaporation	Reflective (infrared), conductor	MEMS	Interconnection, reflective surface in optical MEMS, bonding pad	All
Au–Sn	Evaporation of multilayers, bulk	Low-melting-point alloy	Packaging	Method of package sealing and interconnection	All
$Ba_2Ti_9O_{20}$	Ceramic sintering	High dielectric constant, low TCK	Resonator	High-Frequency band pass filter	Wireless
Cr	Sputtering, evaporation	Intermediate adhesion layer	MEMS	Adhesion layer	All
Cu	Wire drawing	Very low electrical resistivity	Cable	Transmit electrical signals	Cable
Cu	Electrodeposition	Very low electrical resistivity	Integrated circuit interconnects	Carries current between transistors and other electrical components in an integrated circuit	All
Cu	Sputtering, evaporation, plated	Conductor	MEMS	Interconnection	All
Diamond-like carbon	Sputtered, ion-beam, CVD	Hard, wear resistant, corrosion resistant	MEMS	Durable film	Optical
Epoxy	Organic synthesis	UV curable	Modulator	Bonds optical fiber to planar waveguides	Optical
Glasses	Sintering, spin-on	Hermetic	Packaging	Sealant, portal	All
In	Evaporation	Low-melting-point metal	Interconnection, packaging	Solder joints	All
InGaAs	MBE	Small direct bandgap semiconductor	2.5 μm photodetector	Detect light	Optical

Table 2 (*Continued*)

Material	Synthetic Technique	Property	Component	Function	System/Network
InGaAsP	MOCVD	Small direct bandgap semiconductor	Lasers and photodectors	Convert electrical energy to light	Optical
InP	MOCVD	Small direct bandgap semiconductor	Optoelectronic switch	Route optical signals	Optical
$LiNbO_3$	Czochralski growth of single crystals	Electro-optic	Modulator	Imprints RF data stream onto continuous-wave optical signal using optical interference	Optical
$LiNbO_3$	Czochralski growth of single crystals	Electro-optic	Attenuator	Decreases intensity of light propagating in a waveguide	Optical
$LiTaO_3$	Czochralski growth of single crystals	Piezoelectric	Surface acoustic wave	High-frequency filter	Wireless
Methyl methacrylate	Polymerize then drawn	Optically transparent	Plastic optical fiber core	Light waveguide	Optical
Ni	Sputtering, evaporation, plating	Magnetic	MEMS, packaging	Magnetic tranducing, diffusion barrier	All
NiTi	Sputtering	Shape memory alloy	MEMS actuation	Thermal actuation	Optical
Paralenes	Gas-phase polymerization	Low-temperature conformal conductive protective film	Packaging	Protection of electrical circuitry and MEMS devices	Optical
$Pb(Zr,Ti)O_3$	Sol–gel, sputtering	Electro-optic	Modulator, switch	Imprint data stream, switch optical signals between waveguides	Optical
Pd	Plating, sputtering, evaporation	Inert noble metal	High-temperature/power	Interconnection	Wireless
Permalloy (Ni_xFe_y)	Electroplated	Magnetic	Magnetic cores	Magnetic tranducing	MEMS, IC
Photoresist	Spin-on	Photosensitive, structural material	Component fabrication	Masking and etching template, LIGA molds	Optical MEMS
Polyethylene	—	Electrical insulator	Sheath	Provides liquid water barrier and electrical insulation	Cable
Poly-Si	CVD	Resistive, mechanical strength	MEMS structures, integrated circuits	Mechanical element in MEMS	MEMS, optical

Polystyrene	Drawn from preform	Guides light	Plastic optical fiber cladding	Contains light in fiber	
PZT (lead zirconate titanate)	Crystal growth, sputtered	Piezoelectric	MEMS	Actuation	Optical
Si	Crystal growth	Semiconducting, mechanical element	Integrated circuits, MEMS	Conductive path, mechanical structure, substrate	Optical, wireless, MEMS
SiC	CVD	Semiconducting, hard, conductive, stiff, and wear resistant	MEMS	Semiconductor, sensor material, wear resistant	MEMS
Silicone rubbers	Thermally cured process	Conformal protective seal, electrically conductive	Packaging	Encapsulant	Optical, wireless
SiN	CVD	Passivation, mechanical element	MEMS structures	Tailorable passivation, protective layer	Optical, wireless
SiO_2	Sol–gel	Optically transparent	Optical fiber	Confines optical signal	Optical
SiO_2	PECVD (plasma enhanced chemical vapor deposition)	—	Arrayed waveguide grating (AWG)	—	—
SiO_2	MCVD, CVD, thermally grown, spin-on	Preferentially etched stable oxide, thermal and electrical insulation	MEMS structure and integrated circuit material	Electrical and thermal insulation, MEMS sacrificial layer, optical fiber cores	MEMS, IC, optical fiber
SiO_2:Ge	MCVD (modified chemical vapor deposition)	Higher refractive index than pure SiO_2	Core of optical fiber	Carries optical signal	Optical
SiO_2:Ge	MCVD	Photorefractive	Fiber Bragg gratings	Filters single wavelength or modifies pulse characteristics	Optical
TBCCO	Thin-film deposition	Superconducting	Resonator	High-frequency band pass filter	Wireless
Teflon	Polymerized then extruded	Protective sheath	Plastic fibers	Cladding	Optical
Ti	Sputtering, evaporation	Sticks to most surfaces	Metallization	Intermediate adhesion layer	Optical, wireless, MEMS
TiN	CVD, PVD	Passivation protection	MEMS	Diffusion barrier material	Optical, wireless, MEMS
W	Sputtering, evaporation	High-temperature metal	Packaging	High-temperature interconnect, diffusion barrier	All
ZnO	Reactive sputtering	Piezoelectric	MEMS	Transducer material	Optical

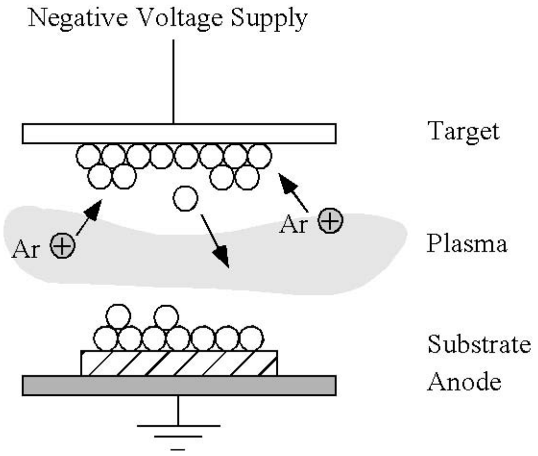

Fig. 17 A simplified schematic of the sputtering process.

ination usually from the filament.[84] In addition, the materials to be evaporated are limited by the temperatures (or power levels) that can be established.

Electron-beam evaporation eliminates these disadvantages and for this reason has become widely used for depositing pure films. In principle, virtually all materials can be evaporated by this type of source.[81] For electron beam evaporation, the evaporant material is placed in a water-cooled crucible. Electrons are thermionically emitted by heated filaments and guided by electromagnets focused into the crucible. The energy from the electrons melts a region of the target. The material evaporates from the source and covers the suspended substrate with a thin film. Commonly used for metallization, wafers mounted above the source are typically rotated to ensure uniform coverage and are sometimes radiantly heated to promote adhesion and uniformity.

4.4 Chemical Vapor Deposition

Chemical vapor deposition (CVD) forms films by thermal decomposition or chemical reaction of gases. The desired materials are deposited directly from the gas phase onto the surface of the substrate. A benefit of this technique is that conformal films can be made; that is, films that coat the entire geometry of the substrate's features. Other physical deposition techniques such as evaporation and sputtering are limited on conformality due to shadowing effects caused by features that block the path of depositing material. Drawbacks of CVD are that high temperatures (several hundred degrees) are required and suitable reactant gases do not always exist. Several materials used in telecommunications are routinely deposited by this technique, including silicon dioxide and silicon nitride. Metals used for interconnects and barrier layers, such as tungsten, tantalum, titanium, and molybdenum, are also deposited by this means.[84] CVD is also a technique of choice to achieve epitaxial semiconductor films. These films are important for semiconductors for photonic devices (laser, photodiodes, and others) as well as microelectronic devices,[85] in which perfect registry is required to reduce loss or attenuation during transmission. A modified form of CVD is used in the development of optical fiber and is discussed in the optical fiber section.

4.5 Epitaxy

The word *epitaxy* is Greek in origin: *epi* means "on" and *taxis* means "arrangement." Specifically, epitaxy concerns itself with the *arrangement* of atoms directly *on* top of other atoms. This technique provides a means of controlling doping profiles and forming single-crystal films so that devices can be optimized. Epitaxial layers can be formed by growth techniques of vapor phase,[81,84,85] liquid phase,[31,86] or molecular beam.[87–89]

In the vapor-phase version, the substrate wafer acts as the seed layer. Growth temperatures in this technique are also considerably lower than the melting point (about 30–50% lower).[85] In it, layers are deposited directly from the gas phase. In a typical reactor, either RF inductive coupling or radiative energy heats the substrates. A carrier gas is flowed over the substrate surface initiating a reaction. Epitaxial layers may also be doped by the adding of impurities to the gas of deposition. In this way, the varying of the partial pressure of the dopant species can control the resistivity. For silicon epitaxy, the growth rate ranges from 0.6 to 1 μm/min.[80]

In the liquid-phase version, the growth of epitaxial layers occurs by direct precipitation of the liquid phase. The reactant solution containing the materials to be deposited cascades the surface of the substrate in a heated environment. The substrate acts as a seed layer for crystallization. Growth rates typically range between 0.1 and 1 μm/min.[84] Liquid-phase epitaxy (LPE) is useful for multilayer films in which precise doping and composition are required. Commonly used for gallium arsenide and other III–V compounds, limitations of this technique include poor thickness uniformity and rough surface morphology. This technique has been replaced by CVD and by molecular beam epitaxy for some applications.[81]

Molecular beam epitaxy (MBE) involves the reaction of one or more thermal beams of atoms with a crystalline surface at ultrahigh vacuum (with pressures of 10^{-10} torr or lower). Substrate temperatures range form 400 to 900°C, and the growth rate is relatively low at 0.001–0.3 μm/min.[84] MBE can precisely control both chemical species and doping profiles with dimension control on the order of atomic layers.[85]

Two types of epitaxial growth may be distinguished: homoepitaxy and heteroepitaxy. Homoepitaxy occurs when the film and substrate are the same materials; for example, epitaxial (epi) Si deposited on a Si substrate. An epi layer is desired because it is freer from defects. In addition, impurities of controlled amounts are also attainable.[90] The importance of this technique arises from the need to control resistivity (by doping levels) and conductivity type (*n* or *p*) of which epitaxial films can be controlled independent of the substrate.[91]

Heteroepitaxy occurs when films and substrates are of different materials, such as AlAs on a GaAs substrate. In these configurations, heterojunctions, or interfaces between two single crystals of differing compositions or doping, are made that create materials of differing bandgaps. These configurations confine charge, which helps achieve a high level of radiative recombination in the active region of the device, thus yielding a high quantum efficiency.[31] Optoelectronic devices such as light-emitting diodes and lasers are based on compound semiconductor heteroepitaxial film structures.[81] Lasers also employ the contrast in electrical properties at heterojunctions to pump electrons into higher states (or invert electron populations)[92] so that when they fall to lower ones, they discharge a photon.

4.6 Crystal Growth

Some semiconducting materials are deposited epitaxially by CVD, while others are grown from melts. In crystal growth,[93] two prominent methods are used: (a) the related techniques including float zone, Bridgman, or Stockbarger, in which the molten material is cooled slowly through a small temperature difference, which causes it to solidify,[90] and (b) the Czochralski method in which a rotating seed crystal is pulled slowly from the melt and in turn grows successive layers frozen at the solid–liquid interface[94] (both are depicted in Fig. 18). One critical requirement for crystal growth by these techniques is that the material must melt congruently, in other words, the melt composition must be the same as the crystal composition. Common materials grown by the Czochralski technique are single crystals of silicon (with a melting point of 1414°C) and gallium arsenide (with a melting point of 1238°C). After growth and surface finishing, the ingot is cut with a diamond tip saw into slices or wafers. These wafers are then evaluated for resistivity, polished, and loaded into plastic trays.

Other crystals important for telecommunications are also developed by this technique. They include semiconductors such as Ge, compound semiconductors[45] such as GaAs, GaP, InAs, InSb, AlSb, and InP, modulator materials such as $LiNbO_3$, and ferrites such as garnets.[91]

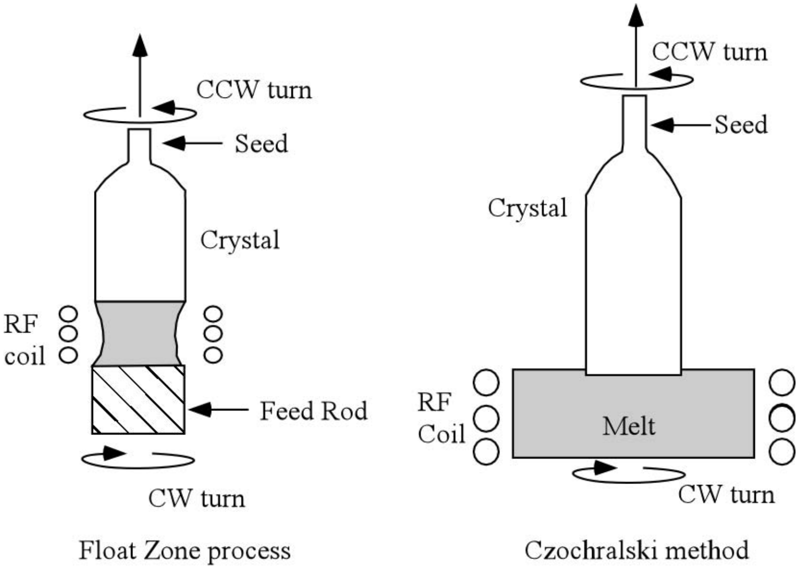

Fig. 18 A schematic of (a) the float zone process and (b) Czochralski crystal pulling apparatus.

4.7 Plating

Electrodeposition is a method of depositing metals and alloys by means of electrochemical reactions. Generally, this deposition technique requires an aqueous solution that contains metal salts. In the process, the metal ions gain an electron (and thus are reduced) and, in turn, are deposited. There are two major approaches: electrolytic and electroless. The main difference is how the electrons used for reduction are supplied. In electrolytic deposition they are supplied by an external source such as a battery or power supply. In electroless plating, they are supplied by a chemical reducing agent in the solution.[91]

Electrolytic deposition is discussed widely.[91] The range of uses of electroless deposition in telecommunications is quite formidable. Several metals[95,96] are commonly plated electrolessly including nickel, copper, cobalt, gold, silver, palladium, platinum, and rhodium. Electroless solutions contain the metal salt, a reducing agent, a buffer (pH adjuster), stability additives for desired properties, and a complexing agent. Because the metal salt and reducing agent are consumed, they must be replenished. This approach is advantageous because both conductive and nonconductive surfaces can be plated. In addition complex shapes can be plated and no special anodic assemblies are needed. Equipment needs are simple as well, for they entail plating tanks and precise temperature regulation. This technique is used in telecommunication power systems such as copper coils for inductors and also in their associated magnetic layers. Electroless deposition is also a means for fiber metallization.[97]

5 VISION OF FUTURE COMMUNICATION COMPONENTS

Imagine walking down Broadway in Manhattan and bumping into a friend from days past. Spontaneity takes over and you want to find a cozy coffee shop to reminisce over a cappuccino. Where's the nearest coffee shop? Do they serve cappuccino? What are their hours?

In addition, with the advent of smart cards and radio-frequency identification (RFID) tags, consumers will be able to walk through the checkout at the bookstore, grocery store, or clothing boutique without having to scan individual items or removing their wallet—the amount will be deduced from your e-money credit or debit card.

Communications technology is already starting to deliver some of these services. Wireless data lend rudimentary access to the Internet and with the aid of the global positioning system (GPS) your precise location can be identified. Grab your cellular phone and call to see if any tables are unoccupied. Violà!

Unfortunately, most of these services are in their infancy and only provided on separate hardware. For example, the Palm VII personal digital assistant allows download of simple website data, and GPS handheld units typically only provide a map centered about a given location.[98] On another revolutionary front, digital cellular phones are being developed with large bandwidth in order to access the Internet. Many telecom companies are developing and implementing third-generation (3G) wireless networks.

This only describes the wireless arena. What could you do if you had a fiber-optic connection in your home? Forget returning movies to the rental store down the street. Download your favorite movie in real time in HDTV wide format for display on a large screen using high-luminosity projectors. The audio and visual quality will surpass that of the movie theater, and you won't have any gum stuck to the bottom of your shoe. The only requirement here is an optical modem, which will be available soon at a reasonable price. Of course, the same fiber will carry voice, data, and video.

What is the vision for materials research? Long-term fundamental research toward controlling the behavior of dopants and defects in solid-state materials will be the challenge for the next several decades. The task of understanding inorganic solids is challenging enough given the thousands of structure types along with variable stoichiometry, but to also consider dopants that encompass most of the periodic table of elements makes the task nearly impossible. Yet, impurities on the order of parts per billion dictate the electronic properties of semiconductors and insulators. With this type of knowledge, engineers could design structures on the atomic scale.

Focusing on short-term research in materials for wireless devices and networks, areas that show promise for future incorporation into telecommunication systems are photonic bandgap antennas, phased array antennas,[99] fuel cells, and soft radio (i.e., further integration such that an entire phone can fit in a package about the size of a credit card).

To improve optical devices and networks, materials research is necessary in the following areas: (1) organic materials have advantages in tailoring at the molecular level and mechanical flexibility as seen in the design of plastic transistors for digital paper,[39] and the major limitation is poor reliability of organic materials due to decomposition from high-intensity light, high temperature, water vapor, and oxygen; and (2) advances in the integration of inorganic materials especially in the form of thin films, where birefringence in waveguides prevents many planar analogs to fiber devices, are needed.

Predictions for future communication systems suggest advances in optical networking to reduce cost of optoelectronic devices and in wireless networks to increase bandwidth, perhaps by utilizing higher frequencies are necessary. Optical networking has yet to take advantage of the integration and miniaturization that has propelled the microelectronics industry. The first step is to incorporate separate components and functionalities into the same package using either free-space optics or waveguide technology as seen in modulator and attenuator packages. Next, integrating these components onto the same substrate or wafer can greatly reduce cost, for example, in electroabsorption modulated lasers (EMLs). Finally, to easily connect fibers to wafers will complete the manufacturing process.[100] The future looks bright for optical communication!

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CHAPTER 44

USING COMPOSITES

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1 INTRODUCTION

When I started working with fiberglass in 1962, many products such as small boats were already in production. Most parts were built by hand labor using rudimentary processes and nonoptimum resin systems.

When boron and carbon fibers became available, they offered the opportunity to compete with aluminum for lightweight stuctures. I was fortunate to be part of an integrated team dedicated to demonstrating the structural capabilities of these fibers by the design, fabrication, and testing of representative aircraft components.

The success of the group led to the opportunity to teach short engineering courses in the United States and Europe. From these experiences I learned that there was a lack of understanding of the interaction between engineering design, materials selection, and manufacturing processes. There were many people who knew what they wanted to achieve but had no understanding how to accomplish their desires.

This chapter is intended to guide the reader through the complex process of conceiving a suitable application for composites and then generating a producible and inspectable design that can be manufactured at a profit. If there are any applicable catch phrases, they would be:

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- To surpass what is presently available, Imagine what is possible!
- Cooperate within the product team to spread the burden of achieving goals of quality and cost.
- Perfection is not common; define what level of “bad” is acceptable.
- Consider failure a learning experience rather than a mistake requiring blame.
- Try to fill a vacuum when there is no one providing your intended product or service. (For example, a recent list of qualified contractors for the restoration of concrete structures with composite patches had no listings for Maine, New Hampshire, and Vermont. Surely there is a need for that service in those states.)

Because it is impossible to predict the specific needs of every reader, I take a broad-brush approach to describe how to convert a designer’s idea into a profitable reality. This chapter discusses materials, processes, and applications to enhance the reader’s understanding of the factors impacting composite design.

The composites industry is divided by product lines, which determine which particular materials and processes are used in a particular plant. Available combinations of resin and fiber are increased by the choice of fiber form: continuous, chopped, the many types of woven fabrics or braided preforms. Processes range from hand labor to sophisticated computer-controlled mechanical marvels. Although the combinations of materials and processes are numerous, much specialization exists because the technologies are too complex for one person, or even one company, to master them all. However, the philosophy underlying a successful composite application is always the same.

2 EVALUATING POTENTIAL PRODUCTS

Composites are truly wonder materials capable of being made into excellent products, but only if they are utilized properly. That is a big “if.” Some notable failures have occurred, such as attempts to use expensive aerospace materials and techniques to replace thin-gauge aluminum sheet metal structures on utility aircraft.

History offers interesting examples of successful composite applications. The ancient Egyptians added straw to the mud used for bricks so that taller watch towers could be built to provide earlier warning of attacks. Assyrian archers increased the range of their arrows by switching from vines and leather for bowstrings to tendons from the legs of antelopes, which are composites of fibrous cells and other cells that provide binding. Tendons have higher strength and stiffness than either vines or leather, which are also composites. And as electricity became more widely used, laminates of cotton cloth and phenolic resins were used as insulators because the laminates were cheap, effective, and easy to make.

The commercial introduction of fiberglass yarns in 1937 led to the establishment of a huge industry with many applications based on the attractive attributes of these materials:

- Pleasure boats and tanks and pipes in corrosive environments based on resistance to water and certain chemicals.
- Printed circuit boards, radomes, and antenna dishes based on low electrical conductivity.
- Filament-wound tanks for firefighters' breathing apparatus based on high ratios of strength and stiffness to weight
- Ducts supplying cooling air to the bays containing electronic systems on military aircraft based on restrictions on part shape. The ducts are the last thing fitted into these cramped spaces and thus must have complex shapes. There is no shape limitation because they are molded on removable cast ceramic tooling. Artificial mountains used in movies are made by draping fiberglass cloth over wooden pegs, spraying on resins, and painting as needed.

Composites offer a wide range of advantages:

- Rather than having to make assemblies from individually made component parts, an automobile manufacturer can mold an entire assembly, such as a press-molded grill panel, which has tabs for attaching lamps and is shaped to match a car's contour.
- Large structures can be made from composites, such as yachts longer than 100 ft. Using continuous processing and tubular fiberglass cloth, liners for leaking water mains have been produced in lengths up to 1400 ft on site.
- Processes exist to produce either a few prototype parts or a large production run. A 40-ft-diameter orange, featured in the Florida building at the 1964 New York World's Fair, was produced by spraying chopped glass fiber and pigmented resin on an inflated war surplus weather balloon, which served as a mold. Corrugated panels used for patio roofs are produced by a continuous pultrusion process in which dry mat and liquid resin are converted to a stack of panels cut to required lengths by an automated system.
- An exotic application is the development of ablative plastics capable of operating at 3000°F for rocket motors. These materials slowly decompose to form an insulating char that contains the gases that provide the thrust for liftoff. High-temperature metals would have been too heavy and require cooling by the evaporation of excess fuel to prevent melting.

The accidental rediscovery in the early 1960s of boron fiber, originally discovered in the 1870s, made possible composites that have significantly higher strength and stiffness-to-weight ratios than metals, especially aluminum, which was important for the aerospace industry. The excellent performance of the F-14 Tomcat was due in part to the large weight reduction in the horizontal stabilizer accruing from the use of boron skins. A low-volume application was the development of expensive rods for dry fly trout fishing. Purchasers can choose from a series of rods with different stiffness and dynamics characteristics, thereby essentially tailoring the rod they select to their casting technique.

In the late 1960s, some 30 companies around the world were attempting to produce graphite fibers with properties similar to the boron fiber. Only 5 suppliers in the United States, the United Kingdom, and Japan successfully produced reproducible graphite fibers. Subsequently, stiffer and stronger graphite fibers were introduced. Recently, inexpensive graphite fibers for commercial applications have reached production levels of several million pounds a year.

Graphite fiber applications were based initially on testing of demonstration aircraft and missile structures by the U.S. military, notably the Air Force Laboratories in Dayton, Ohio. Since 1975, the composite content of military aircraft and helicopters, as well as that of commercial jetliners, has increased steadily. Applications ranging from fighter wings to interior shelves are based on the following material characteristics: (1) ease of tailoring parts to load requirements, (2) high strength-to-weight ratios, (3) no fatigue degradation, (4) no corrosion in service, and (5) high radar absorption.

Initial commercial applications of graphite epoxy fibers used off-grade aerospace fibers with lower stiffness. Applications included golf club shafts, fishing poles, tennis rackets, and drive shafts for pickup trucks. Variation in fiber price negatively affected all these markets, however. The drive shafts performed well, for example, but manufacturers went back to metal based on price. With the introduction of low-cost commercial fibers, these sporting goods applications have rebounded to be a multimillion dollar market.

Newer applications and the reasons for using graphite, now more commonly called carbon, fibers include:

- High-performance race cars (stiffness-to-weight ratio, crash worthiness)
- Rods to stiffen elevated walkways (high modulus)
- Picker sticks to remove drills from oil wells (tensile strength-to-weight ratio)
- Reinforcing corroded bridge columns to bring them back to design strength (stiffness)
- Bows for stringed instruments (stiffness)

Aramid fibers (Kevlar) are lighter than glass and have good tensile properties. Applications include lightly loaded helicopter structures. In a ballistic application, the kinetic energy of a bullet is expended in causing delamination of the armor.

Spectra polypropylene fibers are used in chemical environments where glass fibers are attacked, and in lightweight armor, notably helmets.

An effective nonstructural application uses pads of brittle, high modulus, graphitized pitch fiber in heat sinks to cool densely packed airborne electronic systems. The thermal conductivity to weight ratio of these fibers is five times that of aluminum, and the fibers are 25% lighter.

Reasons why composites are substituted for other materials include:

- To improve a product currently made from other materials by using the unique properties of composites.
    - Fiberglass boats eliminate the need to scrape, caulk, and paint wooden boats every 2 years.
- To provide solutions for problem applications where other materials do not work well or do not work at all.
    - An all-fiberglass shovel was developed to remove snow from electrified railroad tracks.
- To produce luxury items with performance superior to the competition.
    - The Ferrari F-40 had a carbon fiber body produced using techniques applicable to a next-generation fighter plane. It went a little faster, cost $1 million, and met demand by several years production at a low rate to maintain exclusivity.

But once a wonderfully inventive product is conceptualized, reality sets in. The designer must tear himself away from his CAD/NASTRAN computer and join the project team in addressing the constraints in design implementation. These include:

- Production rate
- Limits on product cost
- Availability of processes and facilities
- Capital required for mechanization/automation/enhanced process control systems/quality control facilities
- Environmental issues arising from materials selection

The best planned programs can still have problems that must be resolved by cooperation between team members. Mr. Murphy is usually lurking nearby and can strike at any time. Because we do not know everything about composites, good ideas can turn into unsatisfactory products. For example, radio-frequency (RF) energy is sent from the transmitter of a radar set to an antenna by means of square tubes that must remain precisely the right size over a wide range of temperature. Composites offered significant weight savings over the heavy metal parts made from Invar, but composite waveguides were a failure because their low coefficient of thermal expansion (CTE) was not low enough. However, the CTE data from this effort showed that the CTE of certain graphite epoxy laminates precisely matches that of mirrors of space telescopes. Now graphite/epoxy egg crates support these mirrors in the precise orientation needed despite large temperature swings. Considerable weight savings accrue because massive titanium mounts and thermal strain isolators have been eliminated. Also, Invar tooling is now widely used to produce composite parts to precise dimensions because the tool expands very little as the resin is cooled at elevated temperatures. So new applications sometimes arise from previous failures by understanding what went wrong and using the acquired data in a more suitable application.

3 DIFFERENCES BETWEEN COMPOSITES AND METALS

Composites and metals are different! Designers take care!

Alloy and heat treatment identify metals. Mechanical properties are well defined and maintained during normal processing. In contrast, fiberglass/epoxy can refer to several different types of materials with vastly different properties based on the fiber length and form and the quantity of fibers parallel to the load path. Laminate properties can be varied to match the applied loads by controlling the fiber orientation of tape and fabric plies. Thus, the designer must match the material and process used for his design allowables to that actually used in the product. The required control is obtained by material and process specifications.

Composites are not ductile and do not yield before reaching their breaking strength. Failing strains are much lower than those of metals. The design strain for crossplied graphite/epoxy tape panels is 3000 μin. per inch. Thus, the design must eliminate high point loads such as those introduced by forcing mating parts to fit. Load eccentricities must be avoided to prevent internal ply delamination.

Also, there is a significant interaction between the lay-up process, which is geared to a certain fiber form, and part shape. Fiber form also impacts the mechanical properties used in the design. For example, a small cylindrical tank is best made by filament winding of continuous glass/carbon fibers because high mechanical properties are combined with low cost. Irregularly shaped water lily ponds used by gardeners are buried in earth so the weight of the water is supported. These items are best produced by spraying a chopped glass fiber/polyester resin mix onto the mold. The low cost process is well suited to irregular shapes, and the ponds do not require high mechanical properties.

Lastly, thickness variations of composite parts, typically ±5%, may exceed those of metal parts, especially as part thickness increases. Also, each part can have areas that are either above or below nominal thickness. These variations are due to variations in the weight of resin and fiber per unit area in the materials, and resin movement during processing. Sometimes an extra ply is required to carry the load when minimum part thickness is inadequate. Thickness variations complicate assembly by fastening or bonding because composite parts cannot be forced into shape without damage.

4 MANUFACTURING

As a preview to the major discussion of this chapter, a brief description of the manufacturing operation is desirable. The steps are:

- Clean the mold and apply a release agent to prevent the part from sticking.
- Prepare plies.
- Put the plies on the mold in the specified order (to reduce warpage and obtain optimum mechanical properties).
- Cure the resin by chemical reaction; the resin is transformed from a liquid or paste to a brittle solid.
- Remove the part from the mold.
- Trim, assemble, and paint.

There are three differences from metallic manufacturing:

- Metal parts usually start with constant thickness stock and material is removed in low-stress areas. Composites add plies in high-stress areas to a constant thickness lay-up designed to withstand minimum loads on the part.
- Except for metallic diffusion bonding, it is difficult to produce an assembly in one operation from sheet stock. Composites can be molded easily into assemblies.
- Sandwich construction, especially with skins having several thicknesses, is difficult and expensive to produce in metals. The parts must fit together to obtain a structural bond because only the film adhesive can deform during adhesive curing. However, composite sandwich panels using a variety of core materials are produced in huge quantities by the cure process where the adhesive and skin materials are forced to fit snugly on the core before the resins solidify by curing.

5 THE DESIGN, MANUFACTURING, AND QUALITY CONTROL INTERFACE

A product development team consists of representatives from four departments whose functions are well defined. Management has the responsibility for schedule, cost, and quality and functions as a referee among the other three groups whose objectives and wishes are shown in Fig. 1. Engineering, manufacturing, and quality control usually are strong organizations that are protective of their

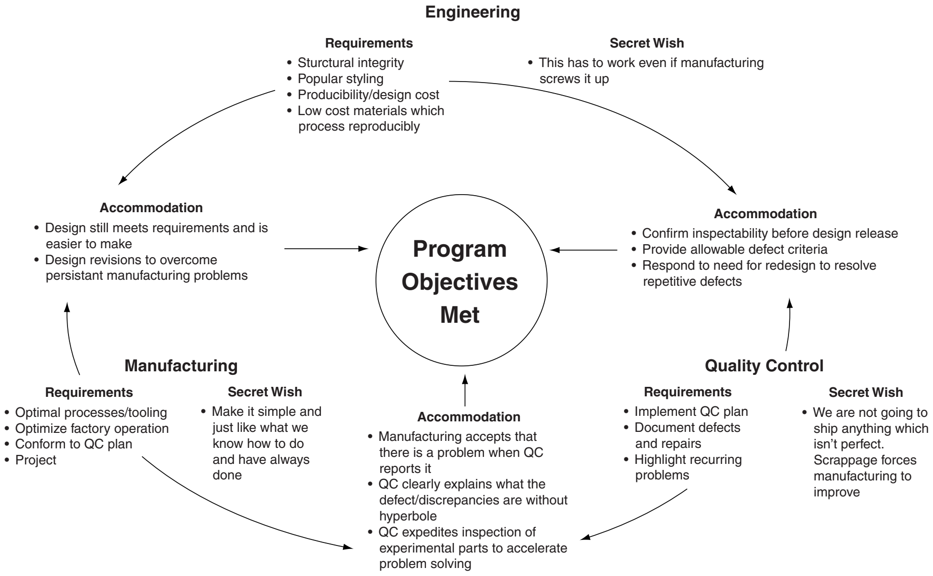

Fig. 1 Design, manufacturing, and quality control interfaces.

function and are provincial in outlook. Their objectives are not always compatible with the needs of the program.

For the success of the program, there must be a sense of give and take to enable each department to achieve its objectives more easily. As an example, a senior vice president at my employer believed in high-performance composites when they were in their infancy in 1966. He directed that an integrated team be formed with all the skills required so that we would work together with these materials under his prodding. Whether it was his directive or the thrill of breaking new ground, each department performed its tasks with a high level of cooperation and concern for other departments' problems. Perhaps that is why we built more types of prototype structures for the first time than anyone else, and they all met the design requirements in full-scale structural and flight tests. Subsequent production programs got off to a rocky start, however, because participants were more interested in protecting their vested interests than following the method of operation used in the development phase.

6 SELECTION OF MATERIAL AND MANUFACTURING CONCEPT

There are four classes of fiber, eight types of matrices, several fiber forms, and eight general manufacturing processes. At first glance, this array of options is bewildering. Often, however, choices are limited. The selection of material and manufacturing process is controlled by design factors such as loading, weight limitations, operating temperature, and perceived value. In addition, customer, government, and industry specifications may apply. Special requirements, such as the increasing use of phenolic materials in trains and aircraft to reduce fire and smoke in case of accident, may apply.

Part shape significantly influences process selection. Fiberglass channels for ladders are a perfect application for pultrusion because they have a constant cross section. Skins for wind turbine blades must have a smooth aerodynamic outer surface, and inner surfaces must fit to the substructure within glue line tolerances. The skins are made by resin injection molding in a closed mold to obtain two controlled surfaces. Resin injection tooling/facilities are less expensive than the alternative of press molding. Parts of complex shape are either vacuum bag or autoclave molded because only one tooled surface is needed for these processes.

Despite these instructions, a designer should take a "clean-sheet" approach and evaluate the factors shown in Fig. 2, which includes both engineering and manufacturing requirements. To assist in material selection, it should be noted that higher properties are obtained from purchased compounds or prepregs. These materials contain matrices that cannot be prepared on the shop floor. If no other data are available, vendor data sheets for these products are a good source of material properties.

At the outset, the intrinsic nature of the various fiber/resin combinations eliminate many possibilities. Service temperature is often the dominant factor. In a process similar to the conversion of ice into slush, the matrix softens due to a thermally induced loss of internal bonding. The softened matrix reduces the ability of the resin to transfer load from one fiber to another. Typically, a very significant decline in laminate properties occurs within a small range of tem-

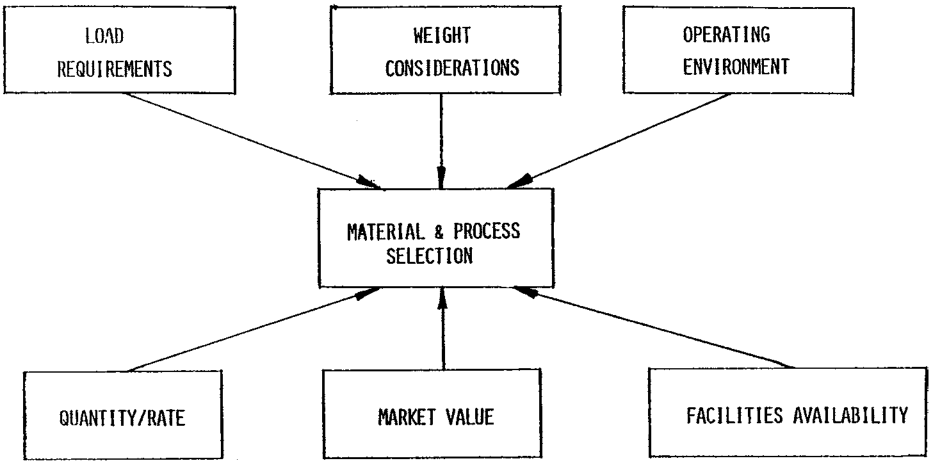

Fig. 2 Factors affecting the selection of materials and processes.

peratures, when the resin is no longer able to function. Typical service temperatures for resins are shown in Table 1.

When service temperature exceeds 150°F for parts having significant applied loads, polyester/vinyl ester type resins are not an option. Epoxy resins have better thermal stability and improve laminate properties. Epoxy resin mixes are of three types: liquid mixes prepared on the shop floor, liquid compounds with better properties prepared by a specialized supplier, and prepreg or molding compounds.

Liquid epoxy resins with enhanced thermal stability are available for filament winding and resin injection parts. In this process, a dry fibrous preform is enclosed in a mold and liquid resin is pumped into the mold to fill the cavity completely. Except for these two cases, all the higher temperature materials use preimpregnated fiber/resin combinations. Difficult-to-handle ingredients can be blended and uniformly applied to the reinforcement to produce unidirectional tape or woven fabric prepregs. For example, a widely used matrix for graphite

Table 1 Typical Service Temperatures for Resins

Matrix	Service Temperature (°F)
Polyester and vinyl ester	150
Epoxy on carbon fibers	260[a]
Epoxy on glass fibers	350
BMI (BisMaleImide)	400
Phenolic	500
Addition polyamide	550
Condensation polyamide	600
Silicone laminating resins	600

[a] Service temperature reduced to compensate for moisture pickup.

fibers contains a waxy, semisolid resin and two powders. The powders are stirred into the molten resin to produce a consistent mix under controlled conditions. Subsequently, the fibers are forced into a sheet of molten resin to form the prepreg. Such a product could never be made on the shop floor.

Weight and load issues are intertwined because the fiber type and fiber form determine how much material is needed to carry loads. Strength-density comparisons for fiber tyes are shown in Table 2.

Fiber form (configuration) has a significant impact on the mechanical properties of the laminate. Although generalizations are risky, some basic rules are:

- Loads are carried by the fiber while the resin transfers load from fiber to fiber.
- Continuous fibers in the load path yield higher properties than short fibers.
- Unidirectional tapes yield higher properties than fabrics do because fabrics have fibers in the off-axes, which may not be needed to carry load.
- Highly loaded, structurally efficient parts have fibers in four directions:
    - longitudinal—main load path
    - 90° transverse—secondary load path
    - 25°–45° off-axis plies in pairs (±)—carry shear
- To control warpage, the lay-up should be symmetrical about a centerline.
- Fibers best carry in-plane loads when flat, so thin fabrics produce higher properties than thick fabrics, due to less fiber waviness or kinking during weaving.
- Thin tapes and fabrics can be accurately matched to the load requirements to produce a lighter but more expensive structure.
- Commercial fibers have fiber bundles 4–16 times as large as the corresponding aerospace grades, so these fibers are preferred for tape and chopped fiber applications where optimum weight is not required.
- Plied laminates have the lowest properties in the thickness direction.

To improve the impact resistance of panels and the joint between the skin and flanges of co-cured stiffeners, an increase in the normal tension strength is

Table 2 Properties of Commonly Used Structural Fibers

Fiber Type	Tensile Strength (ksi)	Tensile Modulus (Msi)	Density (gm/cm^3)
Type E-glass (electrical and structural applications)	500	10.5	2.54
Type S-glass (highest mechanical properties)	650	12.4	2.49
Aramid (Kevlar 49)	550	19	1.45
High-strength graphite, aerospace type (AS-4 or T-300 type)	570	32	1.79
Intermediate modulus graphite, aerospace type (IM-6)	740	40	1.76
Commercial graphite (Panex 33)	550	33	1.81

required. Since stitching the uncured plies puts fibers in the z direction, a mechanical engineer was sent to a sewing machine shop in Manhattan's garment district. He found a deep throat, manually controlled machine formerly used to make shoes and handbags. Kevlar thread with a breaking strength of 150 lb was used to stitch the prepreg prior to cure. In the skin to stiffener flange joints, a stitch was found to be structurally equivalent to a fastener, but much cheaper to install. The development of the resin injection process permits the use of sewn dry fabric, and three-dimensional (3D) woven or braided preforms to produce composites with excellent normal tension properties.

Woven truss core has been incorporated into the walls of large radomes to carry heated air for deicing. Braided structures are integrated assemblies having one or more fiber types intertwined in a 3D configuration. Complex shapes such as sections of an automobile chassis have been produced and have met structural requirements.

The mechanical properties of laminates using various fiber/resin combinations are given in Table 3. The table is intended to illustrate the factors affecting laminate strength and stiffness discussed previously.

However, one additional factor must be considered in selecting design concepts: the panel stiffness requirement, which can be met by using either a solid composite panel or a sandwich structure.

The design and fabrication of composite sandwich panels, with plywood, balsawood, various types of rigid foam or honeycomb cores, is well established. Walls of large truck trailers, made of panels having core and fiberglass plywood skins, have excellent resistance to both corrosion and puncturing. Sandwich panels are widely used in marine and aerospace applications to save weight of panels requiring high stiffness.

Having selected potential materials and configurations, several manufacturing issues must be considered. The first is production rate. Supporting the building

Table 3 Typical Properties of Composites Produced by Different Methods

Process	Material Construction	Tensile Strength (ksi)	Modulus (psi × 100,000)	Density (lb/in.^{3})
Chpped fiber spray	30–50% glass/polyester	9–18	8–18	0.6
Compression mold	SMC 25% glass/polyester	8–12	10	0.76
	SMC 50% glass/polyester	24–30	12	0.65
	50% glass mat/polyester	25–30	9–20	0.66
Filament winding	40% glass/epoxy	80–150	40–90	0.1
Pultrusion	40–80% glass roving/epoxy	60–150	40–60	0.1
	35–50% glass mat/polyester	12–30	10–25	0.8
Autoclave molding prepregs	7781 glass fabric/epoxy	68	34	0.1
	Commercial unidirectional graphite/epoxy	145	100	0.063
	Intermediate modulus unidirectional graphite/epoxy (50% in load path)	180	120	0.063
	Graphite fabric/epoxy	60	70–100	0.063

Note: Data for Comparison Only—Not to be Used in Design of 20 Boeing 737s per month requires a different mind set than producing 30,000 identical parts per month for a popular sedan. The times to complete processes are in Table 4. Data for several fiber-reinforced thermoplastics are included. Their high output rates are widely accepted in automobile design. In contrast, thermosets are limited in high-production applications to press molding of sheet molding or bulk molding compounds because their process times are as short as 2 min.

A second issue, facilities, is very complex. If the facilities, personnel, and experience are available, the project probably will proceed smoothly. Often, equipment, floor space, and utilities must be acquired for a new program at an estimated capital expenditure that is relatively easy to define. The difficulty is in evaluating the benefit to the company. Intangibles include the cost of entering a new business, anticipated profit from the specific operation, stabilizing employment, and possibly using available space. Even the most realistic evaluation is meaningless in times of corporate austerity, or if management is in an expansionist mood and willing to accept risk.

Another option is to buy a complex part with many spec requirements from a specialist who has the expertise, an available facility, and a good track record. In the boom times of the 1970s, a every large aerospace company had a composites shop. Even so, all the precision nose radomes, except for one type, were made in Marion, Virginia, because the supplier there was extremely capable and efficient. However, not all vendors are as competent as this one, a factor that must be included in make or buy decisions.

Table 5 is an overview of composite manufacturing processes. Except for pultrusion, all other processes have a common limitation on part shape: Simply stated, the part must be easily removable from the mold. While it is possible to use segmented (breakaway) tooling, the combination of assembly, disassembly, extra cleaning after use, and higher tool repair costs, constitutes an undesirable cost center. Many parts are made on washout or frangible cast plaster mandrels, simply because the part geometry prevents removal of any other type of tooling. For limited production of a complex shape, a composite costs less than its metal equivalent because the higher cost of the metal forming tools cannot be amortized over enough units. Also, the metal part must be an assembly of individually made components while the composite part can be molded as an assembly.

Table 4 Process Sequence Flow Times

Operation	Time (min)
Pultrude one foot of channel	0.1
Injection mold a small part	0.2
Hot stamp/trim part from TP sheet	0.75
Press mold using rapoid curing polyester SMC	2.0
Filament wind 1# on mandrel	0.1–2.0
Spray chopped fiber/resin to form a bathtub	10.0
Lay-up a small part with fabric prepreg	30.0
Autoclave cure a large panel	540
Hydroclave cure a large rocket nozzle	4800

Table 5 Design Limitations Imposed by Manufacturing Processes

Process/Material	Cure Method	Inherent Restrictions on Design (Not including equipment limitations or special tooling concepts needed to remove parts from mold)	Comments
A. Spray chopped fiber/resin mix on mold (may be supplemented by laid-up mat)	Ambient temperature cure or press molding	● Low properties	● Good for complex shapes ● Low equipment/material cost
B. Hand lay-up of fiber resin combinations on mold (mat, woven yarns and rovings, unitapes, wet resin, or prepregs)	All (determined by resin type)	● Ergometrics ● Elapsed time before resin becomes unworkable	● Labor intensive, but some automation is available
C. Sheet or bulk molding compound (SMC/BMC)	Press molding	● Low properties	● Good for complex shapes and high production rates ● Material costs can be low
D. Resin injection into closed mold or vacuum bag containing reinforcement	Ambient temperature or oven or press cure	● Resin flow limits part size ● Molds must be clamped shut to resist injection pressure	● Good for complex shapes ● Some resins cure quickly ● Compatible with advanced dry fiber preforms such as braided or sewn assemblies that optimize fiber placement in load paths
E. Filament winding of resin impregnated bands of fibers (may be supplemented by local fabric patches)	Ambient temperature or overn cure	● Limited to surface of revolution shapes	● High throughput ● On-site production of lare structures possible ● Moderate startup costs ● Prepregs yield better properties
F. Pultrusion (mat, fabrics, braided preforms)	RF or heat cure in a continuous process	● Limited to constant cross sections ● Design restrictions define configuration/wall thickness	● High output ● Moderate capital requirements ● Braided stock raises properties
G. Automated lay-up of carbon fiber tape/fabric	Autoclave cure	● Tape layers limited to gentle contours ● Fiber placement systems for complex contours ● N/C programs are complex	● Small plies not produced efficiently ● High material utilization of tape ● High capital costs ● N/C programs critical to cost
		● Automated ply cutting and laser projectors to guide ply placement by hand	● Applicable to both tape and fabric ● High output for moderate capital costs ● N/C programs control material cost

Part removal is also facilitated by using a springback factor in right-angle corners (tool 91.5° to get a 90° corner). Other limits on part size are due to the equipment used in the curing process. Heat curing is used for several purposes:

- To rapidly cure the resin as in press molding of SMC (sheet molding compound)/BMC (bulk molding compound) with 2-min cures.
- To cure resins having service temperatures about 200°F, which are almost totally unreactive at ambient temperature.
- To produce low-void composites from dry materials through resin flow into the microscopic gaps between fibers and the plies.
- In these processes, the resin cure requires heating in an oven, press, or autoclave (pressure vessel), which introduces limitations of size and thermal capability.

The last process variable is curing pressure, which is used to densify the laminate by suppressing porosity. Techniques for applying pressure include:

- Contact molding—apply paint or rubber roller to wet lay-up to roll out some of the entrapped air.
- Wrap with shrink tape to apply pressure during oven cure.
- Vacuum bag molding—seal the part with a plastic film and evacuate to 13–15 psi; cure at ambient temperature or in an oven.
- Autoclave molding—place the vacuum-bagged part in a heated pressure vessel and pressurize with inert gas to 45–150 psi (high recurring and facility costs).
- Hydroclave molding—place the vacuum-bagged part in a pressure vessel filled with water. The pressure is developed by thermal expansion (high recurring and facility costs); 250–500 psi.
- Press molding (rapid part production possible; 50–1000 psi.

The last factor in choosing materials and processes is market value, or the perception of what a product is worth. This varies with application. In the highly competitive automobile industry, large volume demands the lowest price possible, and there are few large structural composite applications in production due to cost. The cabs of large trucks, made at the rate of 60,000 per year, trade tooling cost against labor cost, which benefits composites. In contrast, luxury items such as the Corvette, premium tennis rackets, and composite kit airplanes, have a cachet that increases the sales price. As an extreme, high-performance race cars and America's Cup yachts can command a hefty premium over similar products made from conventional materials. Price competition in all areas of the aerospace industry has forced price reductions obtained through production efficiencies, which reduced scrap and rework, and produced cheaper materials that are easier to use.

7 DETAIL DESIGN

An obvious design requirement is that parts must be easily removable from tooling without damage. Design details to be avoided include (1) reverse flanges, which require segmented tooling; (2) molded-in stiffness perpendicular to the direction that a part is removed from a mold, except on the bottom of a box; (3) lack of an adequate taper (draft); and (4) surface buildups, which tend to "hang up" on a mold during part removal.

These principles are illustrated by a press-molded junction box used with buried telephone lines. Flanges to which the access door is attached project beyond the box. The box is molded with the flanges up, so the stiffeners on the opposite face are half-rounds. They are configured to be horizontal to provide maximum support to the vertically positioned terminal strips inside the box. The stiffeners on the remaining four faces of the box are blade configuration, tapering in both width and height away from the flange. The design is tailored to simplify removing the part from the tool. The part is molded in 2 min, then is popped out of the tool quickly.

Five of the eight fabrication schemes involve lay-up of plies on mold, regardless of how the part is cured. In many cases, this operation represents the majority of labor costs. To become more efficient, both the number of plies and the design of individual plies must be optimized. First, the number of plies must be reduced, if possible, by using thicker materials, if they are available. A side issue is weight penalty. In aircraft fiberglass parts, net sections are usually less than 0.1 in. thick, and often are 0.05 inch thick, especially in sandwich construction. For these applications, the preferred dry glass fabric is style 181, which weighs 8.9 oz per yd^2 and yields a composite thickness of 0.01 in. Boat hulls, chemical storage tanks, and swimming pools require thicker laminates and are less weight critical. So woven roving fabrics ranging up to 52 oz per yd^2, with a ply thickness of 0.05 in., are used for these applications. The weight of chopped fiber mat varies from 0.75 to 3.0 oz per yd^2. In addition to reduced labor cost, thicker materials cost less per pound because throughput is increased at the material supplier's facility.

A second issue involves tailoring plies to simple shapes, which can be nested in a cutting pattern, which reduces materials usage. Often, edge plies are used at the edge of panels for bearing strength. They should be cut as strips. For high-rate production, automated ply generation should be considered. Die cutting is applicable for repetitive shapes. In plants producing large numbers of composite parts repetitively, N/C cutting machines are used to cut and identify the plies or ply segments. Then the plies are kitted and sent to the lay-up floor. These N/C ply cutters come from the garment industry. A roll of material up to 60″ wide is unrolled on a 20′ long table. A computer controlled cutter cuts out individual plies of any shape. A second head uses ink to identify each piece. The trick is to locate the pieces close together to avoid wasting material.

A good method to reduce the number of plies is to convert from a monolithic panel to sandwich construction. Several low-cost methods are available.

Flat panels can be laid up and cured in a multicavity press. A widely used system for movable interior partitions used fiberboard skins, paper honeycomb, and film adhesive that cured rapidly. The boat building industry is licensing the patented SCRIMP process developed by Seaman Composites. The process has been used to make individual flat sandwich panels 18 ft × 40 ft, which are sequentially co-cured into a multisided assembly. The skins are polyester-impregnated rovings, and the core is foam slabs prespliced with room temperature curing adhesive. The resin is injected into both skins while the assembly is vacuum bagged. The resin is pressurized in conventional paint spray pots and is uniformly distributed within the lay-up by a proprietary method. The panels have withstood extensive testing with excellent results. The process is attractive because structural panels of any configuration are produced at low material and labor costs with minimal facility requirements.

A third way to produce sandwich panels is co-curing in an autoclave. The core is cut to size, sometimes preformed to the appropriate shape, tapered at the edges if needed. The core is laid over the adhesive and a toolside skin and is itself covered with a second adhesive ply before upper (inner) skin is laid up, a vacuum bag is installed, and the lay-up is cured under positive pressure. For helicopters operating at temperatures below 180°F, the core is a polyamide foam and the panel cure is 1 h at 225°F at 45 psi. The secret of the process is that the skin lay-up, adhesive, and core deform sufficiently under the applied load to fit snugly together to form structural bond lines. Sandwich panels requiring higher loads and service temperatures are produced using nonmetallic honeycomb cores. Precautions must be taken, however, in the tool design and part setup to prevent core crushing from curing loads applied to the edges of the core. These three approaches to sandwich construction all have some provision for localized patch plies, so tapered skins are feasible. In most cases, the core does not have to be processed to accept the extra plies, which provides a significant cost reduction.

The vast majority of composite parts require trimming because they are made oversize intentionally. Generally, it is very difficult to lay up and cure a large part without edge flaws. Unfortunately, composites are very abrasive and soon wear out the high-speed steel/carbide cutters used in aluminum fabrication. The least expensive method of cutting is with a fluid-cooled diamond grit blade. So the preferred part designs have edges that are straight. Cutouts in panels should be minimized, but they can be routed using special design cutters operating at a specific range of revolutions per minute. Initially, cutouts were net molded using tooling to cut the prepreg plies accurately, then the hole was net molded to design configuration during cure. However, improvements in trimming processes have made net molding less attractive.

Waterjet systems used for trimming develop a narrow, focused beam of water at pressures up to 60,000 psi. The water jet travels up to 2800 ft per minute. The high energy of the water jet produces localized failure of the composite to yield a clear-cut edge. The cutting efficiency of the water jet can be improved by incorporating powdered garnet into the stream. Both systems have no restrictions on shape, and may be either manually or computer controlled. Computer-controlled routers are also available that trim panels of any shape efficiently. Both methods are suited to high-rate production. So there are three approaches to trimming, but there is a cost in departing from sawing straight cuts.

Many composite parts go into assemblies such as hulls or decks, bridge repair panels, or major aircraft sections. The two primary methods of joining separate parts are mechanical fastening and structural bonding. The selection of assembly method is limited by the cohesive strength of the available adhesives. At load intensities above the capabilities of the adhesive, fastening is mandatory. Conversely, lightly loaded joints should be bonded due to the complexities of fastening.

Mechanical fastening of composites is more difficult than riveting or bolting metallic structures for several reasons. In addition, there are more problems fastening weight-critical graphite/epoxy structures than fiberglass or Kevlar assemblies. In fact, structural analysis and manufacturing techniques for highly stressed bolted joints were the last major design and manufacturing areas to be fully understood and implemented in production.

As stated previously, composites are very abrasive, so carbide drills are used extensively with special point/flute configurations, often in conjunction with motors that advance the drill a precise amount into the workpiece for each revolution. Even so, many automated drilling systems automatically change drills as often as after preparing only 40 holes. Drilling can produce structural damage around the holes, which can affect structural integrity. This damage includes extensive delamination of the ply where the drill exits the workpiece, localized delamination around the hole, and fiber shredding of the aramid fibers in the hole region.

Hole preparation requires a gentle touch. To further increase the difficulty, many fasteners require a precision sized hole such as 0.188 in. pin requiring a 0.189 to 0.191-in. diameter hole. The countersinks for the fastener heads must be tightly controlled to obtain a good fit so the heads can bear uniformly against the composite.

Fastener selection is a design issue. For fiberglass and Kevlar structures, conventional design fasteners may be used. Stainless steel fasteners are required for wet or corrosive environments.

A whole new family of fasteners is required for graphite/epoxy structures for several reasons. First, the composite is so cathodic that only titanium fasteners are corrosion resistant. A-286 fasteners pit, and monel generates heavy corrosion products that do not appear to reduce joint strength. Also, because carbon fiber composites are more sensitive to high bearing loads than metals, fastener heads, nuts, and washers should be enlarged to provide as much bearing surface as possible to prevent pullout. The larger area also spreads out the high clamping (preload) force desired for good strength and life, so that the laminate is not damaged by matrix crushing. Fastener edge distance and spacing have been studied extensively; $3D$ (D = fastener diameter) edge distance and $5D$ spacing are used widely. Many fasteners are installed with wet sealant as a corrosion preventative. Note of caution: The design of highly loaded joints and the selection of fasteners requires specialized knowledge. Fastener hole preparation and installation also require specific knowledge and preparation of detailed and understandable instructions for mechanics.

The last major problem with assembly of composites is their lack of ductility. Metallic structures can be clamped together to produce an acceptable fit. In one case, an aircraft's wing covers were machined in the flat and forced to fit the curved substructure by hydraulic clamps. Such a procedure is guaranteed to cause severe structural damage to composites by internal delamination. Thus, mating composite parts must be made to fit by shimming with precured composite shims and paste epoxy adhesive that cures at ambient. In lightly loaded joints, the shim adhesive can be reasonably thick if the composite members are overdesigned, so that bearing failure will not occur. In highly loaded joints, the shim thickness is reduced because of bolt flexibility. In graphite/epoxy overlap specimens pulled in tension, the joint strength was reduced 40% when the shim thickness was increased to 0.06 in. from 0.03 in. The 0.125-in. titanium bolts were kinked locally because the shim could not resist the applied load as well as the composite could.

As a result, the aerospace industry has agreed on the following procedure for resolving gaps in bolted joints:

Gap	*Procedure*
0–0.005 in.	Do nothing
0.005–0.03 in.	Use liquid shim
Greater than 0.03 in.	Use solid shim to reduce gap to less than 0.03 in.; then use liquid shim

Good design practice for composite assemblies involves controlling the faying (mating) surfaces by tooling. The substructure of a torque box assembly is usually tooled to control the chordal height of the spar and ribs. If the cover tooling reproduces the substructure contour, the cover will fit reasonably well. The cover thickness variations are thus reflected away from the joint to simply fit up. The mechanical assembly of a complex graphite/epoxy structure is a challenging, difficult, and expensive operation. However, through the design, manufacturing, and quality control interface, it can be made manageable. Also, as the assembly is understood, adjustments and refinements to the design and manufacturing process can be made to expedite production and reduce costs.

The bonding option presents two alternatives that manufacturing prefers to mechanical assembly: (1) structural bonding of individual parts and (2) an integrally molded assembly produced by curing several lay-ups concurrently.

The manufacturing objective for structurally bonded assemblies is to produce an acceptable unit with a minimum of effort with little rework or scrap. To achieve these aims, the process must provide for:

- Cleaning the mating surfaces to prepare them for bonding
- Good fit up of mating surfaces to glue-line tolerances
- Locating the parts in the proper orientation during adhesive cure
- Using a thin fiberglass/nylon cloth (scrim) to guarantee a continuous bond line
- Obtaining a consistent bond-line thickness within specified limits.

The surfaces of individual parts are contaminated with mold release to prevent them from sticking to the mold, dust from trimming, and fingerprints. Surfaces can be prepared either by abrasion followed by a solvent wipe, or removal of a sacrificial external peel ply. Peel ply is preferred for large areas and local areas at the center of a large area. Highly stressed aircraft parts are usually made with a peel ply to avoid damage to the fibers.

Part fit up to bond-line tolerances is mandatory for structural integrity and fatigue resistance. The optimum process consists of a balanced lay-up to prevent warpage and geometrically accurate tooling to control the contour of the faying surfaces. However, these preferences cannot always be granted. Often, for cosmetic purposes, the tool surfaces must be away from the joint, as in the case of boat hulls. Consider the example of a very large ocean-going fiberglass/polyester hull approximately 3.5 in. thick. Thickness variations could be almost ±0.25 in. thick. Using surveyors' techniques, shipyard workers measured the wall-to-wall distance and laid out the trim line on the bulkhead 0.125 in. undersize. The 35 × 30 ft bulkhead was lifted into the hull at the desired station and local interferences sanded until the bulkhead was seated in the desired position. A gusset was cured in place on one face of the bulkhead while it was supported. After cure the scaffold was removed, and the gap between the hull and bulkhead was filled with glass fabric and resin before the other gusset was laminated in place. This is a large-scale example to explain the concept of sanding or patching parts to obtain the proper glue line for the adhesive being used.

A factor in joint strength is width. Peak stresses occur at the edges and decline toward the center. The average allowable stress decreases with length at a certain point because added area contributes little load-carrying capability. While manufacturing prefers narrow joints to ease the fit up problem, engineering also benefits through weight reduction. More importantly, manufacturing prefers bond lines that are in straight-line elements on flat planes. Bonding over bumps is risky due to the high potential for improper bond-line thickness. Antipeel fasteners reduce bond-line stresses in highly stressed or complex-geometry bonded joints.

Two categories of adhesives are used in composite structures:

- Paste or liquid adhesives, typically mixed on the shop floor and cured at ambient (bond-line thickness 0.002–0.02 in.)
- Calendered film adhesives that are cut to size, placed in the joint, and cured at elevated temperature under applied loading (bond-line thickness 0.001–0.01 in.)

The paste adhesive tolerates a thicker glue line with larger tolerances than the film adhesive. Thus, if the analysis, and weight considerations, can tolerate paste adhesives, they are preferred because parts can have larger tolerances in the fit up.

In addition to maintaining an adequate bond line by satisfactory fit up, a thin cloth is used to prevent adhesive squeezeout. Individual parts also must be positioned relative to each other as specified by the engineering drawing. Small assemblies are bonded in a holding fixture. As assemblies become larger, fixtures become more cumbersome. An alternative is to provide coordinating holes in the design so parts can be pinned together during bonding. These holes can be subsequently used for antipeel fasteners, installation of systems, or to make joints fail-safe with structural fasteners.

Despite the precautions needed to assemble bonded structures, bonding is still much easier for manufacturing than fastening.

The concept of co-cured structure is an attempt to eliminate the assembly problems discussed previously. The idea is to place several lay ups on the mold at once to produce a panel. The application to foam sandwich and honeycomb sandwich structure has been discussed previously. Another concept that has been evaluated by the automotive industry involves resin injection. Dry fiber preforms representing several parts are assembled from fabric, mat, braided stock, and knit fibers. Stitching of the dry fabric to enhance normal tension strength is feasible. The preform is placed in a closed mold and resin is injected. After cure, the assembly is removed from the mold. Using resins with isocyanate curatives, an assembly can be made in several minutes. One automaker has combined 50 stamped steel parts assembled by welding into two composite chassis members joined by bolting. I-beam cross-section ribs for military aircraft are molded with at least one attachment clip integral to the web. Each clip saves making and tracking an individual part through the factory, as well as the cost of installing several fasteners.

A common method of providing localized stiffness in commercial fiberglass structures involves placing machined pieces of foam or honeycomb within the lay-up and molding them in. Applications include the keel areas of boats, circumferential rings on composite gasoline storage tanks, and helicopter fuselage panels. Many carbon fiber/epoxy panels are molded with integral stiffeners or attachment angles co-cured on the skin. The tooling setup shown in Fig. 3 was used to mold the panel shown in Fig. 4. The method is suitable for I-blade, tee, hat, and joy stiffeners. The designer can reproduce in composites configurations that have worked well in sheet metal structures. Before committing to a design, however, the designer must understand how much stiffness is required. The cheapest and easiest approach is to add strips of unidirectional material to the skin to react to moderate bending loads. Where bending or flutter is a severe problem, a stiffened panel is required. Stiffener effectivity is greatly enhanced by adding unidirectional plies in the cap to increase the inertia so that the number of stiffeners may be reduced. The most structurally efficient stiffener is the hat section, but mandrel removal is a problem. Unless access to inflatable mandrels

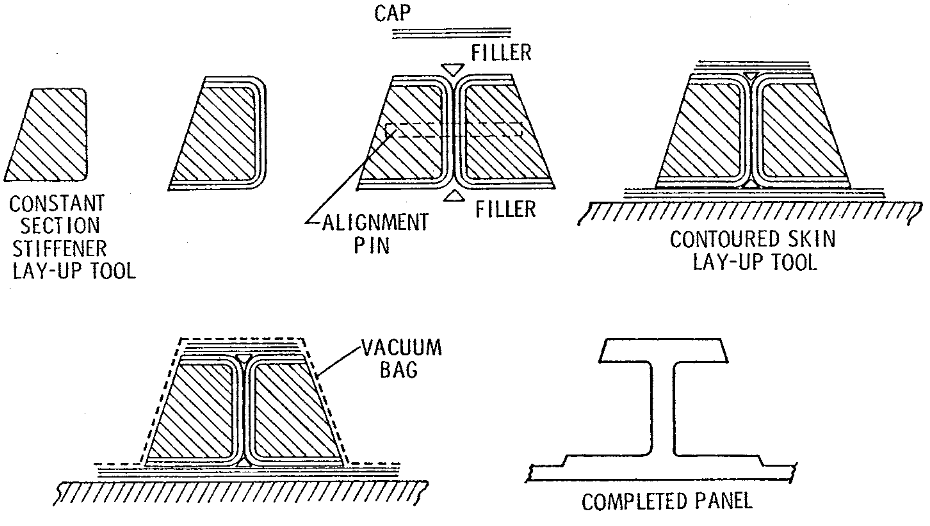

Fig. 3 Co-curing stiffeners with skins.

Fig. 4 Co-cured fusilage skin panel with integral stiffeners and frames.

is available, other stiffener configurations should be strongly considered because they can be molded on reusable tools. The costs of single-use washout or meltout mandrels are high, and no foam withstands the normal autoclave cures at 85 psi and 350°F used to maximize graphite/epoxy laminates properties for 260°F service. Teflon mandrels are surprisingly difficult to extract due to the compressive stress up to 5 ksi exerted by resin shrinkage.

In summary, integrally stiffened panels are both weight and cost effective when compared to comparable fastened structures. However, these benefits can be obtained only by designing panels with a minimum number of stiffeners of a configuration whose mandrels can be removed easily. Fortunately, structures for helicopters or passenger aircraft can be cured at 250°F, so flyaway foam mandrels left in the part are a possibility.

8 PRODUCIBILITY CHECKLIST

With a design concept or two in mind, the designer should review a producibility list to lighten the burden on manufacturing. To meet quality and cost objectives, design, materials selection, and process must be coordinated. In the composites industry, with such varied products, processes, and materials available, generalization is difficult. Thus, the reader is invited to add his or her requirements to this list and eliminate those not pertinent to his or her situation.

The design must provide for low-cost manufacturing, including:

- Contour symmetry so a tool can make multiple part numbers
- Radius of $\frac{1}{8}$ in. on male tools and $\frac{1}{4}$ in. radius on female tools to assist lay-up
- Cheapest materials that can meet all the design objectives, e.g., don't use graphite when glass will do
- Design lay-ups symmetrical about the centerline of the thickness to reduce warpage
- Laying up a minimum number of plies by using thicker materials
- Easily drapable satin weave fabrics for highly contoured parts
- Simplified ply shapes
- Simplified preparation of cores for sandwich structure
- Compatibility with high-rate processes by using automation where possible
- Molding a minimum number of parts
- Minimal curvature to trim
- Compatibility with advanced tooling concepts
- Self-contained curing fixtures
- Removable stiffener mandrels
- Low thermal expansion tools for dimensional accuracy of surfaces
- Ease of part removal
- Tooled surfaces selected to expedite assembly
- Avoid sacrificial, complex, or hard-to-use tools
- Simplified assembly methods
- co-curing sandwich panels
- Co-curing integral joints
- Molding stiffeners on a precured skin with secondary bonded joints
- Thermoplastic molding
- Automated drilling and fastening
- Inspectibility
- Compatibility with NDI systems (if required). NDI stands for nondestructive inspection. X ray or processes similar to CAT scans are used to find flaws inside a part without cutting it up.
- Inspection-critical areas defined
- Allowable defects defined
- Repairs for specific defects documented

Materials selection has a significant effect on a project's success. Factors to be considered include:

- Matrix meets service temperature requirements
- Sufficient resin flow to minimize porosity and enhance ply bonding
- Cure temperature and pressure compatible with application, e.g., expensive autoclave processing of a small boat is not required
- Rapid cure to enhance throughput and minimize the need for multiple tools
- 3D woven or braided dry fabric preforms increase laminate properties in *z* direction
- Reproducibility of batches of material mandatory for part consistency

Lastly, many processes have been optimized for throughput. It is necessary, however, to review the selected process to see if improvements can be made. Relevant issues include:

- Reducing needed workers' skill levels by simplifying operations
- Reducing labor-intensive processes by capital investment, e.g., ceiling-mounted laser projectors that show the location of partial plies eliminate lay-up tooling and save 50% of the labor cost
- Reducing long flow times so that multiple tools are not needed
- Obtaining a tight-tolerance fit of mating surfaces to be joined by bonding or fastening
- Joining many parts by making an integrally molded structure instead of bonding or fastening individual parts
- Compatibility with automated NDI so that erroneous defects can be found

The intent of this section was to highlight various options and pitfalls in composite design and processing. Consultation with experts in the various material categories or processes will further enhance the technical base of the project.

9 ETERNAL QUALITY CONTROL QUESTION

How good do we have to be and how bad can we be to produce a usable part?

The quality control requirements obviously vary according to the end product. A cafeteria tray is press molded in 2 min from dry fiberglass mat, surfacing plies (called veils), and a cupful of liquid resin. Every part is inspected visually, and a few are probably structurally tested initially. Periodically, the weight of the ingredients and trays, and operating parameters of the press, are checked so that the product that was tested is produced for the entire production run. Any parts that are too difficult to repair are scrapped. There is a limit on how much attention can be paid to a cheap product with low structural requirements.

In contrast, manufacturing an advanced composite (CFRP) aircraft structure is monitored closely. A former chairman of the FAA Composites Committee was very outspoken that Aunt Minnie should be able to arrive in Peoria as safely in a composites aircraft as she did in a metal aircraft. Resolving his concern is a two-step process, with the first stage including:

- Materials procured to rigid specifications, with properties defined by allowables
- Verification of design points by the test of coupons made from the specified material
- Manufacturing process controlled by a detailed process specification
- Additional design verification by testing elements and subcomponents produced using both specifications
- Static and fatigue testing of full-scale articles produced as described above

At this point, the capability of the specified material and process to produce a structure that meets all of an aircraft's requirements has been verified. For the production department, the intent is to produce exactly the same product, or a better product, hundreds of times over a period of several decades, thereby verifying the design. To ensure meeting this safety requirement, a detailed inspection plan (Fig. 5) must be implemented. The system works well until a defect is found. The engineering staff must evaluate the effect of the flaw and determine the disposition of the part: use as is, repair per a specified procedure, or scrap. Many programs now have documentation covering allowable defects, standard repairs, and mandatory scraps to reduce the engineering effort with regard to disposition of a defective part. So the engineering staff must first design an optimum part, then documentation must be prepared to define how "bad" it can be and still meet design requirements. Standard repairs for repetitive defects must be provided.

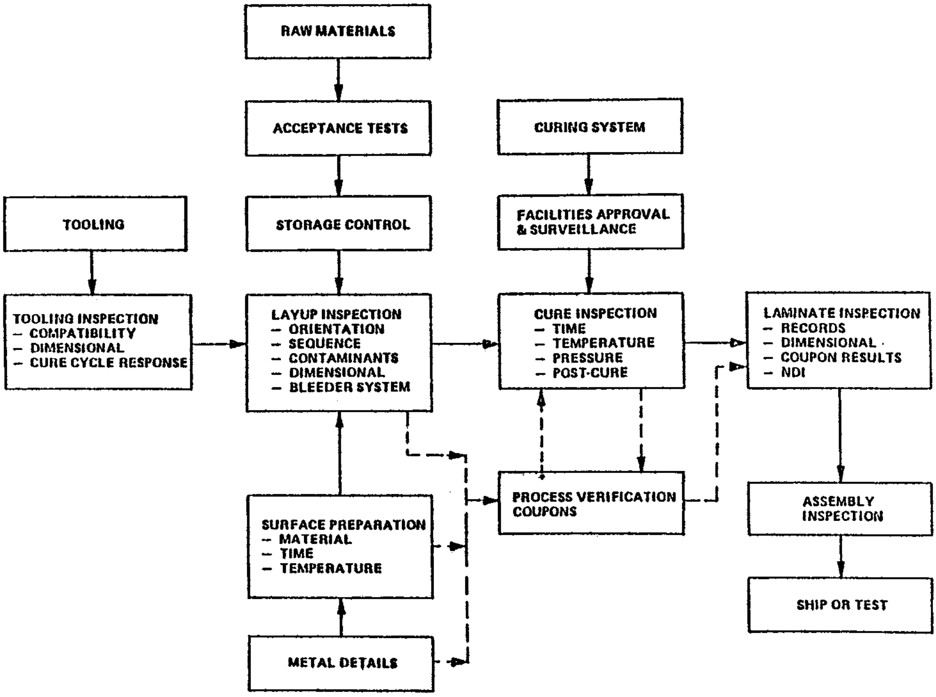

Fig. 5 Inspection requirements for flight-critical structure.

This concept also applies to other industries. While evaluating the processing of large fiberglass boat hulls, for example, I have observed mechanics making repairs based on the engineering department's sketches and instructions. Other flaws were left as is.

In the case of press-molded parts, a visual examination of the part surface and tap testing for porosity may be sufficient.

In summary, every product requires a quality control plan suited to the product's function and its value. As structural requirements and costs increase, the engineering design effort must address defects. Parts with unacceptable discrepancies or inadequate repairs must not be placed in service. Conversely, usable parts should not be scrapped unnecessarily.

For lightly loaded consumer products, the engineering department can relax as the manufacturing department struggles to always make the product look pretty and keep defects within tolerable limits.

10 ENVIRONMENTAL CONCERNS

A composite manufacturing plant inherently has many environmental problems simply due to the nature of the materials and processes used. Under prodding by the Environmental Protection Agency (EPA), industrial hygiene has improved significantly since the mid-1980s. Even so, profits from a well-conceived production effort can be lost due to EPA penalties. So environmental concerns must be a part of the materials selection and design effort, as well as a requirement for manufacturing facilities. There are four problem areas: airborne vapors, skin contact with allergens, dust, and disposal of waste.

Many resins contain ingredients with low boiling points. Polyester and vinyl ester contain styrene whose odor permeates even well-ventilated facilities. Phenolic resins have the sweet odor of unreacted phenol and give off formaldehyde. A group of mechanics working on a highly classified project were awarded compensation for medical problems because, reportedly, they were working in an inadequately ventilated area and absorbed some chemicals.

An early curative for epoxy resin mixes prepared on the shop floor required melting prior to use. Brown fumes were produced. I experienced them. They stained our clothes (I was wearing "epoxy" workpants) and anything else nearby. The first curative for polyurethane castings also required melting and was a carcinogen. Both compounds where withdrawn after years of use.

Epoxy resins produce dermatitis once the body becomes sensitized. My best technician did not develop a rash until 17 years of exposure, but I have seen new employees develop rashes in several months. The only cure seems to be to avoid contact with epoxy materials prior to cure and with dust from cured panels. Material suppliers are required to provide materials safety data sheets listing the ingredients in generic terms. These sheets should be carefully reviewed by the safety team before new materials are introduced to production. In well-ventilated areas, in my opinion, problems, except for dermatitis, are not significant as long as normal precautions are taken. Food and drinks, for example, are no longer allowed in lay-up areas.

Chips are produced when metals are drilled or cut. Composite machining, on the other hand, produces dust. Recently, water jet cutting of composites, which does not produce dust, has been introduced into production. Also, water-cooled drills produce a gritty slurry instead of dust. Maximizing the use of water-cooled drilling in modern equipment is cost effective and provides a free safety benefit. Dust can be controlled by using vacuum hoses next to cutters, spray booth filters, and vacuum cleaners to replace brooms.

The EPA has guidelines for the disposal of solvents and paints. Composite scrap and dust should be buried. This is especially true for carbon/graphite composites. Once, in Cleveland, scrap carbon fibers were incinerated and the residue was blown into a transformer that shorted out. Much of the city was dark until repairs could be made.

Even though a history of effective methods to prevent environmental problems exists, using new materials or inattention can cause problems. So, to reduce environmental hazards in the factory, the designer should confer with the safety department before transmitting a design to the production department.

CHAPTER 45

COMPOSITES IN CONSTRUCTION

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1 INTRODUCTION

1.1 General

For the past few decades, the aerospace industry was the major user for fiber-reinforced polymer (FRP) composite materials. Recently, civil engineers and the construction industry began to realize the potential of these materials in providing remedies for many problems associated with the deterioration and corrosion of infrastructures.

Civil engineers have dealt with different types of composite materials for decades, including wood (natural composites), plywood (laminated natural composites), and concrete (particulate composites). Polymer composites are ‘‘engineered’’ materials that encompass a wide range of materials where two or more, physically distinct and mechanically separable, components are combined together to form a new material that possesses properties that are notably different from those of its individual constituents. The primary load-carrying component is the fibers, while the matrix acts as a binder, an environmental protector, and stress distribution phase of the laminate. Fibers are available in different types, grades, and shapes. Typical types of structural fibers include glass (E, S, AR grades), aramid (Kevlar), and carbon fibers with different grades. Two types of polymers are available, namely thermoplastic and thermoset polymers. In most of the structural application, thermoset resins are preferred. Examples of ther-

Handbook of Materials Selection. Edited by Myer Kutz

moset resins include epoxies, unsaturated polyesters, vinylesters, aminos, phenolics, and urethane resins.

1.2 Advantages

Some of the attractive and unique features of these materials are their durability and resistance to the marine environment, their toughness, particularly at low temperatures, their vibration damping capabilities, their energy absorption under earthquake loading, their electromagnetic transparency, their low value of coefficient of thermal expansion, pigmentability and decorative characteristics, and their high strength-to-weight ratio. These unique properties can be used to produce an optimum structural system with minimum life-cycle cost, fabrication and construction cost, and time.

2 CONSTRUCTION APPLICATIONS OF COMPOSITES

Applications where composite materials can show their superiority over other conventional materials are discussed below.

2.1 Aggressive Environments

- Waterfront structures
- Water and wastewater treatment plants structural elements
- Water declination plants

Fig. 1 Seismic upgrade of columns using composites. [Courtesy of Structural Composites Construction Inc. (SCCI)]

- Off-shore structures (off-shore oil rig platforms, marine risers, etc.)
- Cooling towers
- Petrochemical and nuclear power plants
- Paper and pulp mills
- Chimneys
- Pipes

2.2 Repair and Retrofit Infrastructure Systems

Due to their unique properties, composites can provide structural engineers with the answers to many structural problems. The two major applications are seismic repair and rehabilitation (Fig. 1), and corrosion repair (Fig. 2). These apply to buildings, bridges, and other infrastructure systems. Composites can also be used to upgrade the structural performance and capacity of reinforced concrete, steel, and wood and masonry structural members inland and off-shore. In this process, several laminates of composites are bonded to the finished surfaces of the structural member in the specified directions. In early 1990s, the majority of the applications focused on the ductility enhancement of concrete columns, especially in seismic areas such as California. In this particular application, the fibers

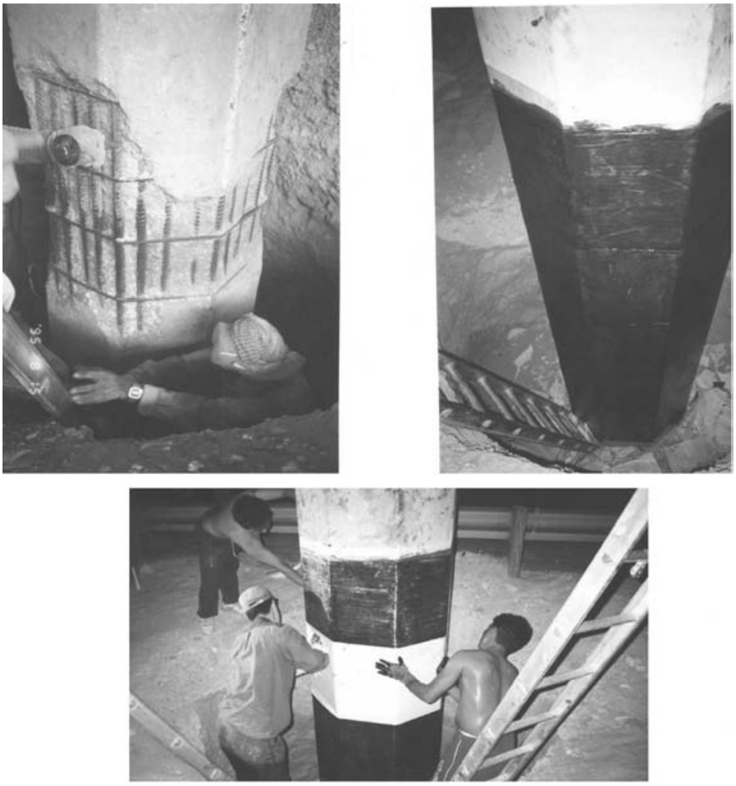


Fig. 2 Reinforced concrete corroded column repair applications using composites. (Courtesy of Sigma Composites, LLC)

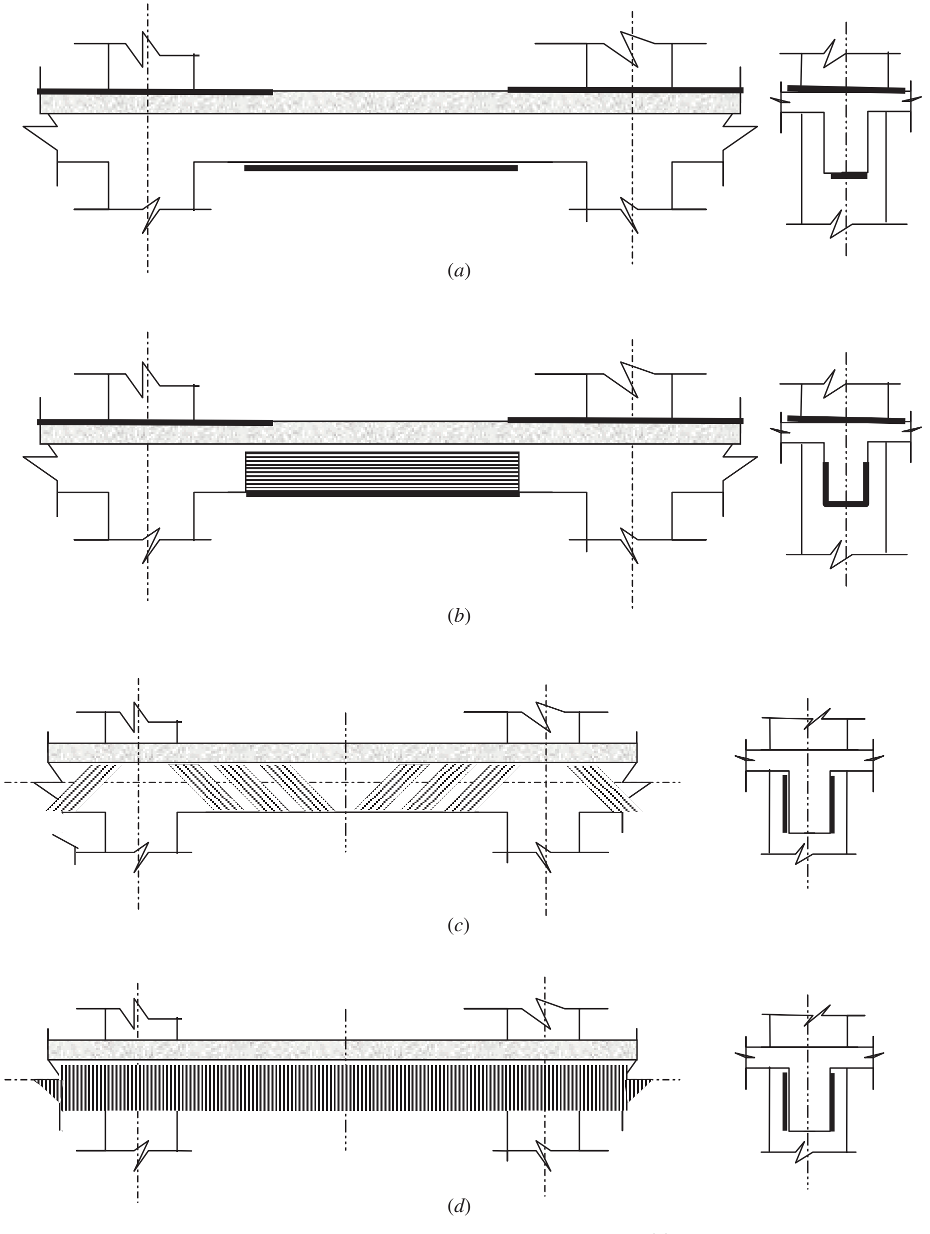
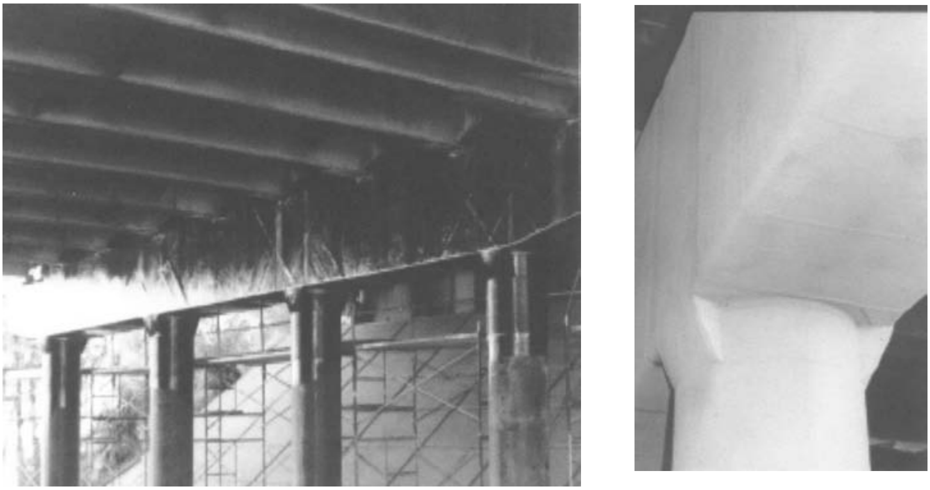


Fig. 3 Typical and the preferred FRP external flexural and shear: (*a*) Flexural reinforcement of continuous RC floor beam using flat $(0°)_n$ laminates schedule; (*b*) Flexural reinforcement of continuous RC floor beam using the preferred U-shaped $(0°)_n$ laminates schedule; (*c*) Shear reinforcement of continuous RC floor beam using inclined laminated strips; (*d*) Flexural reinforcement of continuous RC floor beam using the preferred continuous flat $(90°)_n$ laminates schedule.

are exposed to tension due to the Poisson effect, which, in turn, provide the required hoop stresses without adding to the column's stiffness (i.e., stiffness/strength decoupling). This application has been extended to cover other reinforced concrete structural members, such as beams (flexure, shear, and torsion as shown in Fig. 3), slabs, beam–column joints, and walls. Figure 4*a* shows a photograph of the U.S. Interstate 80 bridge over State Street in Salt Lake City, Utah, that was seismically retrofitted with polymer composites. The bridge consists of four reinforced concrete bents, each bent having four columns, and a bent cap supporting composite welded girders is shown in Fig. 4*b*. A seismic retrofit design was developed using carbon fiber-reinforced polymer (CFRP) composites (Pantelides et al., 2001a) to improve the displacement ductility of the bridge. The retrofit included column jacketing, as well as wrapping of the bent cap and bent cap–column joints for confinement, flexural strength, and shear strength increase. Special provisions were developed for the specifications of the CFRP composite retrofit of State Street Bridge (Pantelides et al., 2001b). The CFRP composite retrofit was implemented in the period 2000–2001.

Some of the potential repair and retrofit applications are:

1. Strengthening of reinforced concrete columns (refer to Figs. 1 and 2, beams (Fig. 5), floor and bridge deck slabs (Fig. 6), and frame connections (Fig. 7)
2. Strengthening of concrete and steel fluid tanks (refer to Fig. 8)
3. Strengthening of stacks or chimneys (Fig. 9)
4. Reinforced concrete shear walls (Fig. 10)
5. Strengthening of slabs-on-grade (Fig. 11)
6. Strengthening of concrete, and steel pipes (Figs. 12 and 13)



(*a*) (*b*)

Fig. 4 (a) State Street Bridge bent in Salt Lake City after being seismically retrofitted with CFRP composites. (b) Detail of column-bent cap joint retrofitted with CFRP composites. (Courtesy Professor C. Pantelides, University of Utah)

Fig. 5 Reinforced concrete beam strengthening applications using precured composite strips. [Courtesy of Structural Composites Construction Inc. (SCCI)]

7. Strengthening of utility wooden poles (Fig. 14)
8. Strengthening of wooden beams and columns and plywood shear walls (Fig. 15)
9. Strengthening of reinforced and unreinforced masonry walls (Fig. 16)

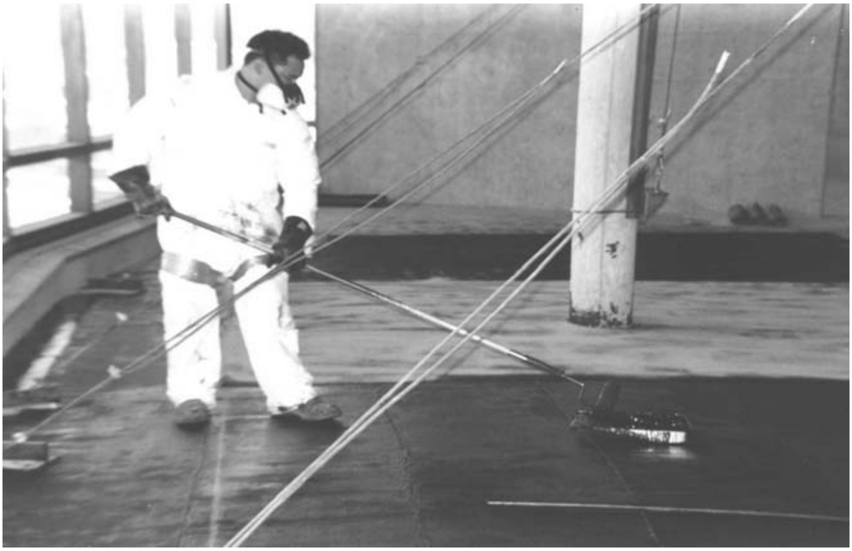
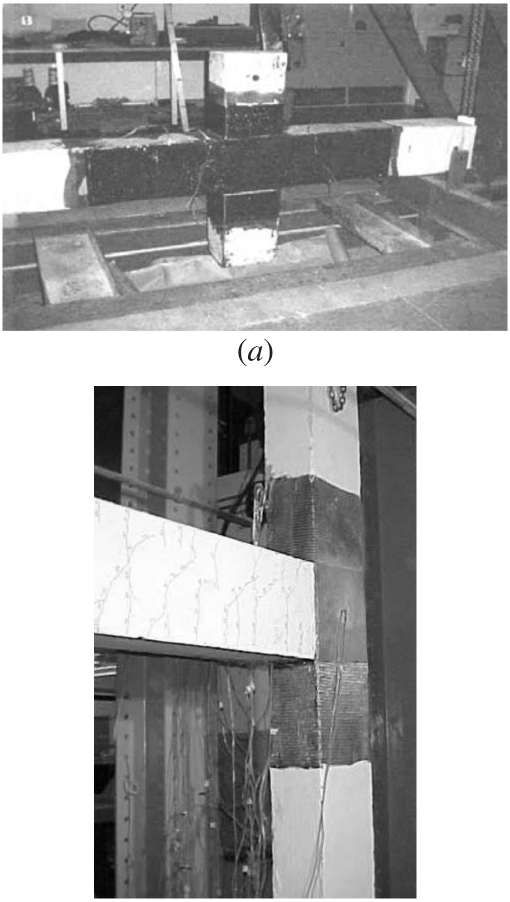


Fig. 6 Reinforced concrete floor slab strengthening applications using composites. [Courtesy of Structural Composites Construction Inc. (SCCI)]


(*a*)

(*b*)

Fig. 7 Reinforced concrete beam-column connection repair applications using composites. (a) California State University at Fullerton, and (b) McMaster University, Canada (Ghobarah and Said, 2001).

10. Strengthening of tunnels (Fig. 17)

11. Strengthening of concrete members for explosion resistance (Fig. 18)

Types of Composite Repair Systems

Currently, the composite repair methods/systems that are available, include:

Wet/Hand Lay-up. In this method, the fibers are in the form of either unidirectional or multidirectional sheets, waived, or stitched fabrics. After surface pretreatment of the structural member, and the application of a thin film of low-viscosity epoxy-based primary, the saturated fibers are applied by hand to the location indicated in the engineering drawings (Fig. 19). The preferred method of saturating the fibers is using custom-designed impregnator (or saturator) to ensure proper and complete fiber impregnation with the resin system (Fig. 20). However, some systems use brush and rollers to wet their fiber materials. The common type of matrix used in wet layup repair application is room temperature

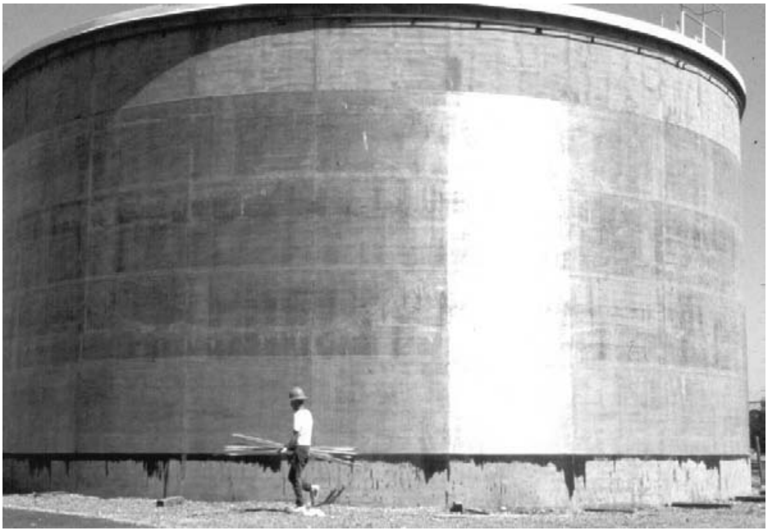


Fig. 8 Confinement of reinforced concrete tank using polymer composites. (Courtesy of Fyfe Co. LLC)

cure two-part epoxy systems. Several types of fibers are being used in this process including standard and high-modulus carbon fibers, glass fibers (including types E, S, and AR). A very limited commercial products uses aramid due to its sensitivity to wet environment that is unavoidable in construction applications. However, it should be noted that aramid-based composites can also be used in this application, provided that the fibers are completely protected from

Fig. 9 Structural strengthening of chimneys using polymer composites. (Courtesy of Fyfe Co. LLC)

Fig. 10 Applications of composites for strengthening reinforced concrete shear walls. [Courtesy of Structural Composites Construction Inc. (SCCI)]

Fig. 11 Structural strengthening of unreinforced concrete slab-on-grade using polymer composites. [Courtesy of Structural Composites Construction Inc. (SCCI)]

Fig. 12 Strengthening of reinforced concrete pipes using polymer composites. [Courtesy of Structural Composites Construction Inc. (SCCI)]

Fig. 13 Seismic strengthening of steel pipe joints using polymer composites. [Courtesy of Professor O'Rourke, Cornell University (Tutuncu, 2001)]

Fig. 14 Structural strengthening of utility wooden poles using polymer composites. (Courtesy of Fyfe Co. LLC)

Fig. 15 Structural strengthening of glue-lam wooden beams using carbon/epoxy sandwich composites. (Courtesy of Sigma Composites, LLC)

the surrounding environment particularly from moisture. Other FRP composites repair systems made of hybrid materials have been used in a number of applications, especially when carbon-based composites are used around metallic parts. In this case, a thin film or a thin mat of E-glass is used to avoid the development of the corrosion process due to the galvanic action created when carbon-based composites are in direct contact with metallic parts in the presence of an electrolyte, such as water, that activates the galvanic process.

Table 1 presents average mechanical and physical values for common fibers for composites used in construction repair applications as compared to steel. The laminate properties are always lower than the dry fiber properties due to the presence of the matrix, which has negligible structural capacity. In addition, if

Fig. 16 Strengthening of unreinforced masonry walls using polymer composites. (Courtesy of Fyfe Co. LLC)

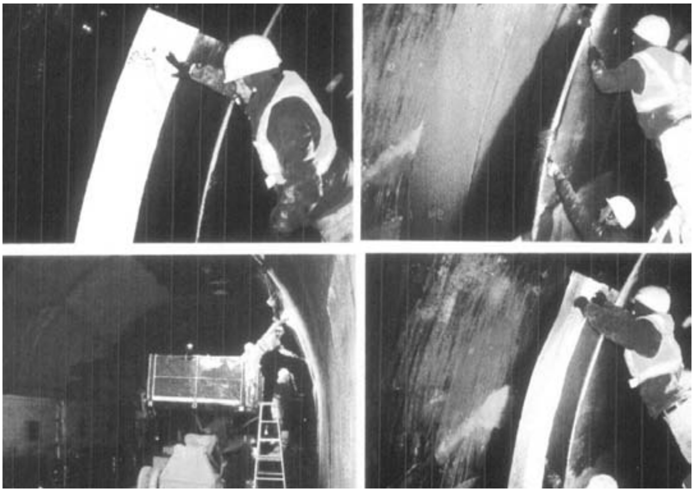


Fig. 17 Strengthening of tunnels using polymer composites. (Courtesy of TONEN Corp.)

Fig. 18 Blast-resistance enhancement of reinforced concrete structures. [Courtesy of Structural Composites Construction Inc. (SCCI)]

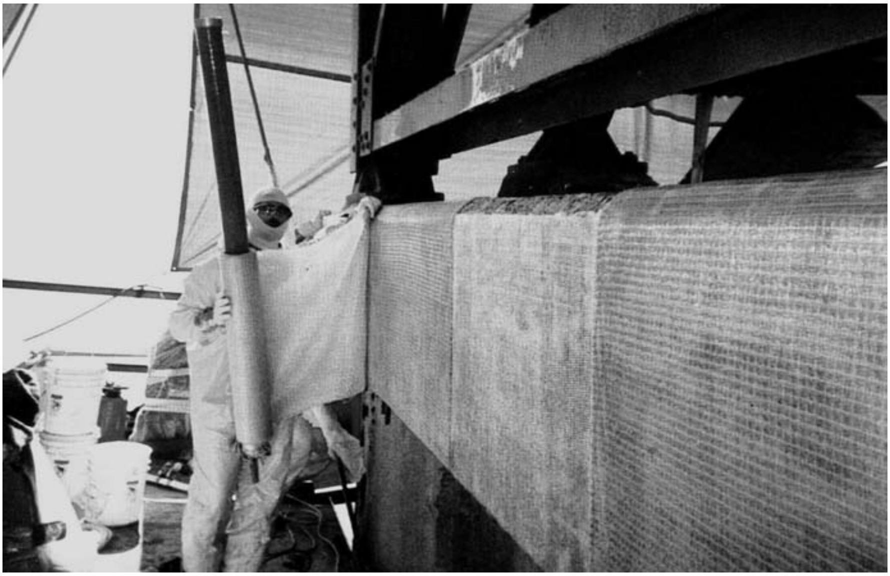


Fig. 19 Wet/hand lay-up repair process. (Courtesy of Fyfe Co. LLC)

the fibers are directed in different directions (off-axis), the uniaxial or properties parallel to the direction is expected to decrease depending upon the plies angles and the volume of fibers in each direction relative to the major fiber direction (on-axis) as shown in Fig. 21. This issue is very important and should be very clear for the civil engineer who is unfamiliar with composites. The structural engineer must distinguish between the fibers and laminate properties when designing a repair system. The most critical information that is used in the design

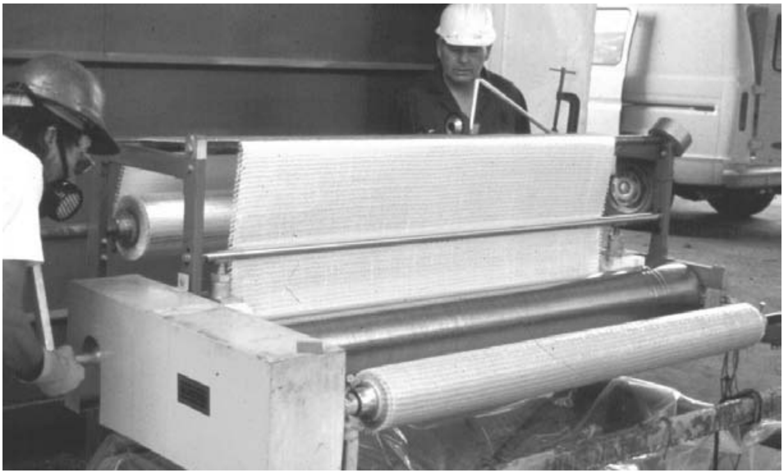


Fig. 20 Use of automated saturators is the preferred method for wet lay-up repair process. (Courtesy of Fyfe Co. LLC)

Table 1 Average Mechanical and Physical Values for Common Fibers for Composites Used in Construction Repair Applications as Compared to Steel

Fiber Type	Average Tensile Strength		Average Tensile Modulus		Density	Average Elongation (%)
	MPa	ksi	GPa	Msi	g/cm^2 (lb/in.^{3})	
E-glass	3450	500	72.50	10.50	2.54 (0.092)	>4.7
S-glass	4480	650	85.60	12.40	2.49 (0.09)	>5.2
Carbon	4825	700	228	33	1.80 (0.065)	>1.3
Aramid	3800	550	131	19	1.45 (0.052)	>2.5
Steel (AISI 1025)	394	57	207	30	7.80 (0.282)	0.12[a]

[a] Yield strain.

is the "laminate" rather than "fibers" properties. Fibers and matrix properties can also be used to predict some laminate mechanical properties, and these results can be used to confirm the uniaxial FRP composite laminates properties supplied by the manufacturer. In this case the civil engineer should have the following information:

- E_{1f} = longitudinal fiber modulus
- E_m = Longitudinal matrix modulus
- ν_{12f} = Longitudinal Poisson ratio of the fibers
- ν_{12m} = Longitudinal Poisson ratio of the matrix
- V_f = fiber volume ratio
- V_m = matrix volume ratio

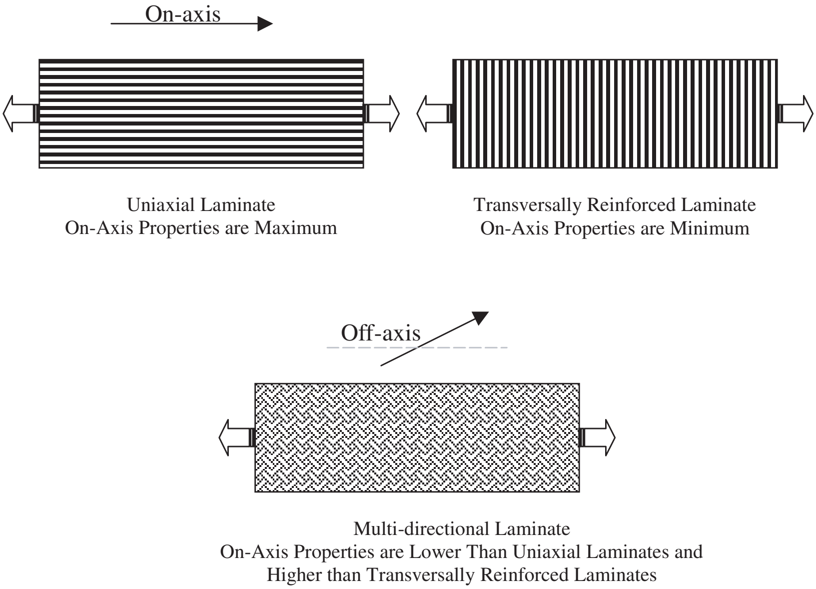


Fig. 21 On-axis and off-axis mechanical properties of composite laminates.

The fiber and matrix volume fractions or ratios, V_f and V_m, can expressed as:

$$V_f = \frac{\text{Volume of fibers}}{\text{Volume of composite}} \tag{1}$$

$$V_m = \frac{\text{Volume of matrix}}{\text{Volume of composite}} \tag{2}$$

Knowing the ratios, V_f and V_m, the void volume ratio can be calculated as:

$$V_\nu = 1 - V_m - V_f = \frac{\text{Volume of voids}}{\text{Volume of composite}} \tag{3}$$

The FRP laminate longitudinal and transversal mechanical properties can be predicted using the following simple expressions (commonly called "rule of mixtures"):

$$E_1 = V_f E_{1f} + V_m E_m \tag{4}$$

$$\nu_{12} = V_f \nu_{12f} + V_m \nu_m \tag{5}$$

$$E_2 = \frac{E_{2f} E_m}{V_f E_m + V_m E_{2f}} \tag{6}$$

where E_1 = laminate longitudinal (major) modulus
E_2 = laminate transverse modulus
ν_{12} = laminate longitudinal (major) Poisson ratio

For simple unidirectional laminate tensile strength predictions, two cases should be considered:

1. If ultimate tensile fiber strain (ε_f^u) is lower than ultimate tensile matrix strain (ε_m^u), i.e.,

$$\varepsilon_f^u < \varepsilon_m^u \tag{7}$$

Accordingly, the laminate failure will occur when the composite laminate strain reaches the tensile fiber strain. In this scenario, the laminate tensile strength can be expressed by the following simple formula:

$$\sigma_t^u = \sigma_{tf}^u V_f + E_m \varepsilon_f^u V_m \tag{8}$$

Assuming a composite laminate with relatively stiff fibers as compared to the matrix, i.e., when $E_f >>> E_m$, Eq. 8 can be simplified further as:

$$\sigma_t^u \cong \sigma_{tf}^u V_f \tag{9}$$

where σ_t^u = laminate longitudinal tensile strength
σ_{tf}^u = fibers longitudinal tensile strength

2. If ultimate tensile matrix strain (ε_m^u) is lower than ultimate tensile fiber strain (ε_f^u), i.e.,

$$\varepsilon_m^u < \varepsilon_f^u \tag{10}$$

In this case, the laminate failure will occur when the composite laminate strain reaches the tensile matrix strain. The laminate tensile strength can approximately be calculated using the following simple formula, which does not account for the statistical distribution of fiber and matrix strengths:

$$\sigma_t^u \cong \sigma_{tm}^u (V_f \xi + V_m) \tag{11}$$

where σ_{tm}^u = matrix l tensile strength
ξ = fiber/matrix stiffness ratio (modular ratio) given by:

$$\xi = \frac{Ef}{E_m} \tag{12}$$

Table 2 presents some average mechanical values for typical FRP composites repair systems. It should be noted that these values are based on 50% volume fraction. In reality, and especially for wet lay-up field applications, the typical expected volume fraction ranges from 35 to 45%. For this reason, the values presented in Table 2 may be reduced accordingly using Eqs. 4–12. It is strongly recommended that the civil engineer require random sampling and American Society for Testing and Materials (ASTM) tensile coupon tests from different patches mixed at the site in order to verify the design-based mechanical properties and to allow for design modifications based on actual field properties of the FRP composite system. A comprehensive document describing these procedures, called AC178 (ICBO, 2001a), has recently been approved and published by the International Conference of Building Officials (ICBO). The engineer is

Table 2 Average Unidirectional Composite Laminate Room Temperature Mechanical Values for Typical Composite Repair Systems Used in Construction Applications

Laminate Composition	Typical Laminate Longitudinal Tensile Strength, σ_{11}		Typical Laminate Longitudinal Tensile Modulus, E_{11}		Typical Laminate Transversal Tensile Modulus, E_{22}		Average Laminate Elongation (Rupture Strain)
	MPa	ksi	GPa	Msi	GPa	Msi	%
E-glass/epoxy	950	138	34.0	5.0	8.3	1.2	2.7
S-glass/epoxy	1100	160	41.3	6.0	8.9	1.3	2.7
Carbon/epoxy	1400	200	138.0	20	10.3	1.5	1.20
Aramid/Epoxy	1300	189	65.0	9.5	5.5	0.8	2.0

Note: Based on 50% Volume Fraction; *Fiber:Resin Volumetric Ratio 1:1* advised to review this important document during the design process as well as during the construction phase for quality control and quality assurance of the composite repair system.

Preimpregnated (Prepreg) Composite Systems. Preimpregnated laminates (prepreg system) are also available commercially for construction repair applications. In this case, dry fabrics are preimpregnated with resin at the controlled shop conditions. Unlike the wet lay-up system, where the composite laminates are fabricated and cured at the site, the prepegs are fabricated at the shop and cured at the construction site. Prepreg composite repair system requires heat blankets for curing the prepreg laminates, which is one of the disadvantages of this system, especially at remote areas and for complex geometry of the structural members to be repaired. In addition, the useful life of such systems is limited and dependant on the storage environmental conditions.

Prefabricated Composite Laminates or Shells. In this method, the fibers are in the form of either unidirectional strips, shells, or sandwich panels. In the case of flat members such as beams and slabs, prefabricated unidirectional composite strips are bonded to the specified locations using epoxy after surface treatment. The common manufacturing process for the prefabricated composite flat strips is called ‘‘pultrusion,’’ which is a continuous process that will be explained later in this chapter. However, several other manufacturing processes are also available for fabricating these strips including press molding, resin transfer molding, and others. In all cases, the average fiber volume fraction for commercially available composite strips is about 65%. The procured unidirectional composite laminates are commonly delivered to the construction site in the form of large flat stock or coiled on a roll for thin laminates. A peel ply is preferred when the surfaces are pretreated to ensure clean bond surface at the time of application.

In applying the prefabricated strips, sanding or removal of the outmost matrix-rich layer is performed to ensure sufficient bondline strength between the composites and the concrete surface. To verify the bondline strength, a pullout field test is often required by the engineer of record (refer to Fig. 22).

For columns repair, prefabricated shells with majority of fibers in the hoop direction are used. After surface preparation, a thin coat of epoxy is applied, and the shell is placed at the required location per the engineering drawings. Straps are used to squeeze out any excess resin (Fig. 23). It is critical that the split lines be staggered with a phase angle of 90°.

Automated Machine Lamination. In this method, the fibers are either dry or preimpregnated. Thermal blanket or mobile curing oven is usually used to achieve the complete cure of the composite laminate (Fig. 24). Following the initial curing process, textured urethane-based paint is hand applied over the cured laminates, which provides ultraviolet (UV) protection of the composites.

Design Considerations for FRP Composite Repair

One of the major issues that the structural engineer should clearly identify is the state of the existing underdesigned or diffident member. This includes the existing and expected future loads, as well as the extent of damage and/or

Fig. 22 Prefabricated composite laminate R/C slab applications and on-site bondline strength test. [Courtesy of Structural Composites Construction Inc. (SCCI)]

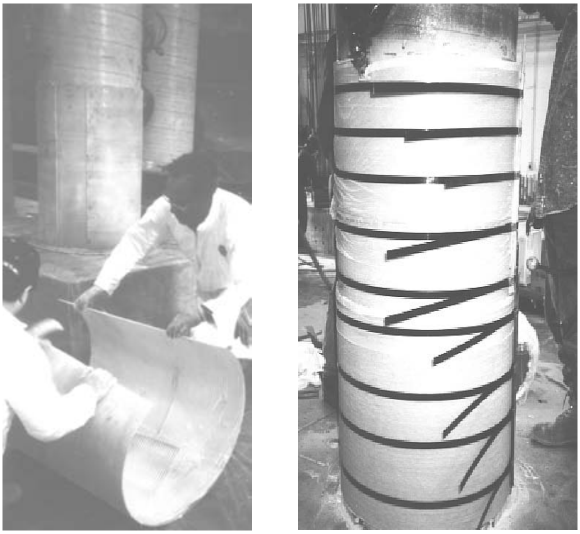


Fig. 23 Hard shell precured composite retrofit system. (Courtesy of Professor M. Haroun, University of California, Irvine)

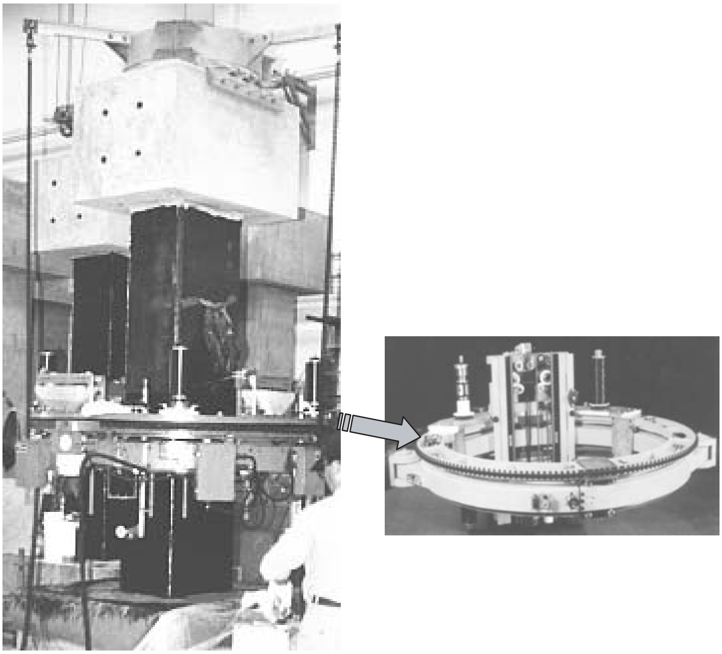


Fig. 24 Automated machine lay-up for bridge column retrofit applications. (Courtesy of Professor M. Haroun, University of California, Irvine)

structural deficiency of the structural members. This includes the residual strength of the concrete that can be determined by testing random core samples from the member to be repaired to instigate the feasibly of using the composites, as well as to determine the type and required specifications for the resin (for wet lay-up systems) or the adhesive (for prefabricated systems) and the type and viscosity of the primer to be used prior to the application of the composite laminates. The next step is the identification of the environmental exposure of the structure. This step is very critical in the selection process for fibers, resin system, required additives (e.g., the requirements of adding UV inhibitors for outdoor applications and fire-retardant additives for indoor applications), as well as the preferred fabrication process of the composites. The structural engineer should identify the locations of damages, for example, in a reinforced concrete member, the locations and quantity of the damaged steel reinforcements should be defined in order to calculate the structural demands for different types of stresses using the appropriate FRP composite repair system. Also, the engineer should quantify the limit state for her/his design in order to specify the efficient composite repair system.

For example, if the main concern were the loss of stiffness, the carbon-based composites would be the preferred choice in this case. On the other hand, if the ductility enhancement for seismic applications is the design objective, glass-based FRP composites would be appropriate to the inherent lower longitudinal stiffness modulus of the materials. Of course, both materials can be used, but the question is the efficiency and the reliability of the system, which is the sole responsibility of the engineer of record. Another issue that should be analyzed beforehand is the creep rupture effects of the FRP composite repair system, particularly for both glass-based and aramid-based composite systems. In this case, a knockdown factor, relatively higher than that for carbon-based composites, should be used to avoid any potential failure due to the exposure to sustained loading conditions. In determining these knockdown factors, several parameters should be considered, including the stiffness and strength degradation due to the exposure to aggressive environments, as well as the strain rate and the type of application. For example, composite jackets used for ductility enhancement, often called “contact-critical” application, will undergo a light strain level until the application of the seismic forces. In this case, a lower creep rupture (or static fatigue) knockdown factor may be used. On the other hand, a composite system applied at the bottom surface of a reinforced concrete beam, often called “bond-critical” application, especially if the a cumber has been introduced to the beam before complete cure of the composites, will be exposed to a relatively higher state of strains. In this case, a relatively higher creep rupture knockdown factor should consider. In this particular example, the carbon-based composite system is the preferred choice.

Design Philosophy of FRP Composite Repair of Reinforced Concrete and Masonry Structures

Flexural Capacity Upgrade of Concrete Members. As mentioned earlier, one of the major tasks in designing with FRP composites is the strength assessment of both concrete and steel reinforcements as well as the present stress conditions. The major design criterion for FRP repair of reinforced concrete structures is based upon the strain compatibility principles. FRP composites have different thermomechanical properties as compared to concrete. Upon loading, the strain developed in the concrete, steel, and composites are assumed to follow a linear pattern (refer to Fig. 25).

Following the ACI318 ultimate strength code procedure, the factored moment should be larger than or equal to the ultimate bending moment of the section, i.e.,

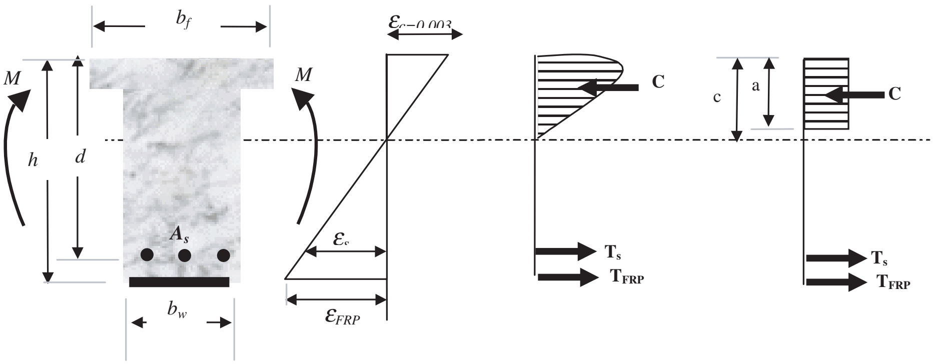


Fig. 25 Distribution of flexural strain and stresses at ultimate for concrete beam reinforced internally with tension steel and externally with FRP composite laminate(s).

$$\phi M_n \geq M_u \tag{13}$$

where ϕ = strength reduction factor, which depends on ductility of ultimate mode of failure and type of stresses (if failure is ductile, i.e., internal steel yielded at time of failure of FRP composites, typical value of 0.9 can be used, otherwise value should be reduced to account for brittleness of ultimate failure)
M_n = nominal or predicted moment
M_u = ultimate moment capacity of the section

The strain compatibility condition can be derived using the linear strain distribution in Fig. 25, and assuming

- the ultimate strain of concrete in compression is 0.003,
- the tensile strength of concrete in tension is ignored, and
- strain distribution along the depth of the beam is linear,

then

$$\varepsilon_{\mathrm{FRP}} = 0.003\left(\frac{h-c}{c}\right) \leq \omega\varepsilon_{\mathrm{FRP}}^{\mathrm{ult}} \qquad (\omega < 1) \tag{14}$$

Here:

- The reduction factor ω is used to prevent delamination of bondline failure. For design purposes, this factor should be taken less than unity and the upper bound depends on the type of the FRP system.
- The strain developed in the laminate at ultimate, $\varepsilon_{\mathrm{FRP}}$, may be reduced if the existing member is exposed to existing service loads, which generates another existing strain component $\varepsilon_{\mathrm{ext}}$ (refer to Fig. 26). In this case,

$$\varepsilon_{\mathrm{FRP}} = 0.003\left(\frac{h-c}{c}\right) - \varepsilon_{\mathrm{ext}} \leq \omega\varepsilon_{\mathrm{FRP}}^{\mathrm{ult}} \tag{15}$$

- The term $\varepsilon_{\mathrm{FRB}}^{\mathrm{ult}}$ is the ultimate or rupture strain of the FRP composites.

Based on the strain compatibility condition and the ACI318-99 procedure (ACI, 1999), the ultimate moment capacity of the strengthened section is given by

$$M_n = A_s f_x(d - 0.5a) + A_{\mathrm{FRP}} f_{\mathrm{FRP}}(h - 0.5a) \tag{16}$$

where A_s = area of tension steel
f_s = stress in steel at ultimate
f_{FRP} = stress in the FRP laminates

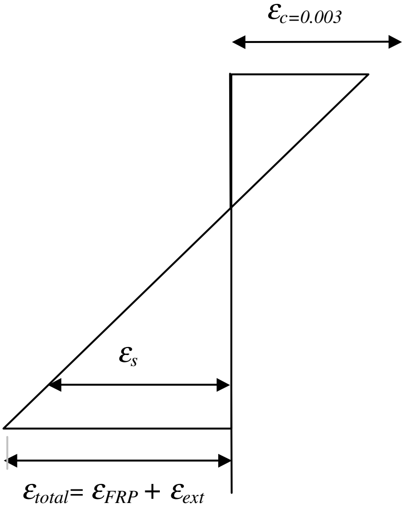


Fig. 26 Total tensile flexural strain when FRP repair applied while the member is loaded.

$a = \beta_1 c$
β_1 = ratio of the average concrete compressive stress to the maximum stress. For concrete compressive stresses f'_c, the factor β_1 shall be taken as

$$\beta_1 = 0.85 \quad \text{for} \leq 4000 \text{ psi} \tag{17}$$

$$\beta_1 = 1.05 - 0.05\,\frac{f'_c}{1{,}000} \quad \text{for 4000–8000 psi} \tag{18}$$

$$\beta_1 = 0.65 \quad \text{for} > 8000 \text{ psi} \tag{19}$$

Similar procedure for predicting the flexural strength of FRP externally reinforced concrete and masonry members is described in the ICBO AC125 (2001) document. According to ICBO AC125 (ICBO, 2001), the flexural strength gain (the component T_{FRP} in Fig. 22, which is referred to ΔF in the Eq. 20) can be calculated using the following equation:

$$\Delta F = \frac{t_f \cos^2\theta f_{jf}}{\text{unit} - \text{width}} \tag{20}$$

where t_f = thickness of the FRP laminates
θ = angle of fiber direction to member axis
f_{jf} = confining strength of FRP composites calculated by following equation:

$$f_{jf} = E_f \varepsilon_f \cos^2\theta \leq \lambda f_{uj} \tag{21}$$

where E_f = tensile modulus of elasticity of FRP composites
ε_f = composite material strain at designated strength
λ = strength reduction factor dependent on type of composites.

This factor is taken as 0.75 for all composites in the original ICBO AC125 [ICBO, 2001b, equation (1), section 7.3.2.1]; however, it is strongly recommended to have different values for different composite repair materials to avoid creep rupture in the polymeric composite laminate(s). Table 3 presents the recommended values of λ for different FRP composite systems.

Detailed information on design procedures can be found in Nanni and Gold (1998), Mosallam et al. (2000), and ICBO AC125 (ICBO, 2001b). Analytical modeling of special applications of concrete slabs retrofit with composites is reported by Mosalam and Mosallam (2001).

Minimum Bond Strength Requirements. For repair applications where the structural performance of the composite system depends largely on the bondline strength of the composites to the concrete or masonry (often called bond-critical applications such as beams, slabs, and walls), the ICBO AC125 (ICBO, 2001b) requires that under ultimate flexural strength conditions, the bond stress developed between the composites and concrete or masonry rate of change shall not exceed:

$$u_u = \frac{d(t_f f_j)}{dx} \leq 0.75 f_t \tag{22}$$

where u_u = bond stress between FRP composite laminates and concrete or masonry
t_f = composite laminate thickness
f_j = laminate stress
x = direction parallel to the fibers

The term $d(t_f f_j)/dx$ describes the rate of change of the fibers net force $(t_f f_j)$ with respect to the distance (x) parallel to the major fiber direction. Equation 22 should be evaluated at sections where this rate is maximum, which is normally at the ends where maximum shear stresses exist. For comprehensive coverage

Table 3 Recommended Values of λ (in Eq. 21) for Different FRP Composite Systems[a]

FRP Composite System	Recommended Reduction Factor, λ
Carbon/epoxy	0.50
E-glass/epoxy	0.30
Aramid/epoxy	0.35

[a] These values are based on room temperature environment. For higher service temperatures and/or severe environments, these values shall be reduced.

of this subject, the reader is referred to two design textbooks, one by Hollaway and Head (2001) and one by Hollaway and Leeming (1999).

Shear and Torsional Strengths Upgrade. As mentioned earlier, the shear strength of reinforced and unreinforced concrete and masonry members can be upgraded using external FRP composite laminates. The previous procedures dealing with flexural strength upgrade assumed that the engineer has checked the shear capacity of the member, and if the member is deficient in shear, additional FRP laminates should be applied. Studies on the torsion straightening of reinforced concrete beams are scarce. The first pilot study on confirming the validity of upgrading the torsional capacity of reinforced concrete rectangular beams using FRP laminates was reported recently by Ghobarah et al. (2001).

Comprehensive studies on the use of FRP laminates to increase the shear capacity of reinforced concrete columns and beams are reported by Haroun et al. (1999) and Kachlakev et al. (2000), respectively.

Circular Sections. According to ICBO AC125 (ICBO, 2001b), the nominal shear strength gain for circular reinforced concrete members of diameter D is given by

$$V_{sj} = 2.25 t_f f_j D \sin^2\theta \tag{23}$$

where V_{sj} = shear strength enhancement provided by FRP composite laminates, lb (N)
t_f = composite laminate thickness
f_j = allowable laminate stress = $0.004E_j \leq \lambda f_{uj}$
λ = reduction factor (refer to Table 3)
E_j = composite laminate tensile modulus
f_{uj} = ultimate tensile strength of the composite laminate
θ = angle of fiber orientation relative to the member axis

Rectangular Concrete Beams or Columns Sections. According to ICBO AC125 (ICBO, 2001b), the estimated shear strength gain for rectangular concrete cross sections or a depth H, parallel to the direction of the applied shear load is given by

$$V_{sj} = 2.86 t_f\, f_j\, h \sin^2\theta \qquad \theta \geq 75^\circ \tag{24}$$

where V_{sj} = shear strength enhancement provided by FRP composite laminates, lb (N)
t_f = composite laminate thickness
f_j = allowable laminate stress = $0.004E_j \leq \lambda f_{uj}$
λ = reduction factor (refer to Table 3)
E_j = composite laminate tensile modulus
f_{uj} = ultimate tensile strength of the composite laminate
H = cross-sectional dimension parallel to the applied shear force
θ = angle of fiber orientation relative to the member axis

For the composites to perform effectively, the corner of rectangular or square concrete section must be rounded using mechanical grinders or other appropriate techniques to a minimum radius of 0.75 in. (20 mm) before the application of the FRP laminate to the pretreated concrete surfaces (Fig. 27).

Rectangular Masonry Wall Sections. According to section 7 of the ICBO AC125 (ICBO, 2001b), the nominal shear strength gain for rectangular masonry wall sections of depth H parallel to the applied shear load is given by

$$V_{sj} = 2t_f\, f_j\, H \sin^2 \theta \tag{25}$$

for composites applied at two sides

$$V_{sj} = 0.75t_f\, f_j\, H \sin^2 \theta \tag{26}$$

for composites applied at one side only with $\theta \geq 75°$. Equations 25 and 26 assume that adequate anchorage is provided by bonding to the wall ends. In addition, it is recommended to use a special anchoring system between the composites and the wall and foundation using metallic or composite connectors to ensure effective shear transfer. It is also recommended to introduce appropriate shear strength gain reduction factors.

Axial Load Capacity Upgrade. The axial (in-plane) capacity of the concrete or masonry member can be upgraded by applying FRP composites in the direction of the applied force. For concrete members, no data is available to confirm this application. However, for masonry walls, numerous large-scale test results indicated that an appreciable gain in the in-plane capacity of the wall members could be achieved by adding fibers in the directions of the applied in-plane loads.

The common method of increasing the axial capacity of concrete members such as columns is by applying the fibers in the transverse (hoop) direction. In this case the composite laminates are subjected to tensile stresses due to the Poisson effect. A large number of research studies were conducted on the behavior of reinforced concrete columns with FRP composite jackets. An early

Fig. 27 Minimum radius of 0.75 in. (20 mm) for noncircular columns is required before the application of FRP composites. (Courtesy of Professor M. Haroun, University of California, Irvine)

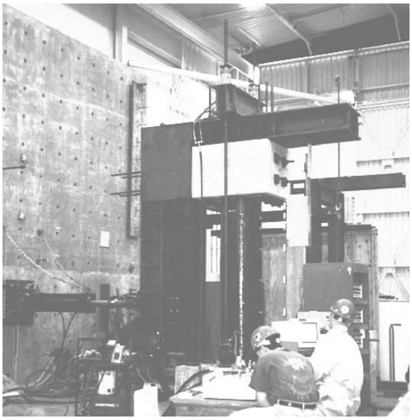


Fig. 28 Caltrans large-scale testing of highway bridge column with FRP jackets. (Courtesy of Professor M. Haroun, University of California, Irvine)

study was reported by Priestley et al. (1992) on the use of E-glass/epoxy composite jackets for seismic retrofit reinforced concrete columns. Another study was conducted by Xiao et al. (1995) on large-scale columns retrofitted with precured shell composite laminates. A comprehensive report prepared for the California Department of Transportation (Caltrans) on retrofitting reinforced concrete bridge columns using different composite systems was presented by Haroun et al. (1999). In this report, both circular and rectangular large-scale columns were evaluated for different retrofit applications including lap-splice enhancement, shear enhancement, and flexural enhancement (Figs. 28 and 29). Currently, a comprehensive experimental and theoretical program is conducted as a joint research project between the University of California at Irvine and California State University at Fullerton. In this program a total of 110 large-

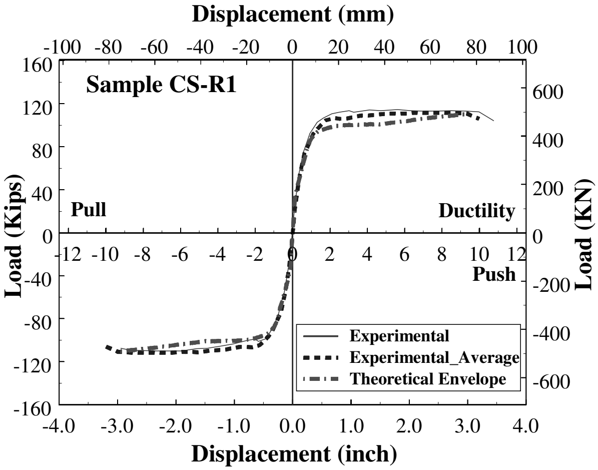


Fig. 29 Theoretical and experimental load–displacement envelope for a large-scale reinforced column with composite jacket (Elsanadedy, 2001).

scale reinforced and reinforced concrete column specimens with different cross-sectional areas including rectangular, square, circular, hexagonal, and octagonal have been tested (Fig. 30). A sample of the experimental stress–strain relations for confined and unconfined columns is shown in Fig. 31. Tables 4 and 5 present summaries for full-scale test results of rectangular, hexagonal, and octagonal column specimens tested in this program. A description of the testing program is presented by Haroun et al. (2000).

Design Procedures. The ICBO AC125 (ICBO, 2001b) has established analytical procedures to predict the strength gain of concrete members transversally reinforced with FRP composite jackets. For circular columns, an equation based on the Mander model (Mander et al., 1988) is adopted. The equation requires that the aspect ratio of the repaired column cross section does not exceed 1.5, otherwise a special analysis is required. According to section 7 of the ICBO AC125 (ICBO, 2001b), the concrete confined compressive strength, f'_{cc}, of a *circular column* jacketed with composites in the hoop direction is given by

Fig. 30 Samples of reinforced and unreinforced column specimens (Youssef, 2001).

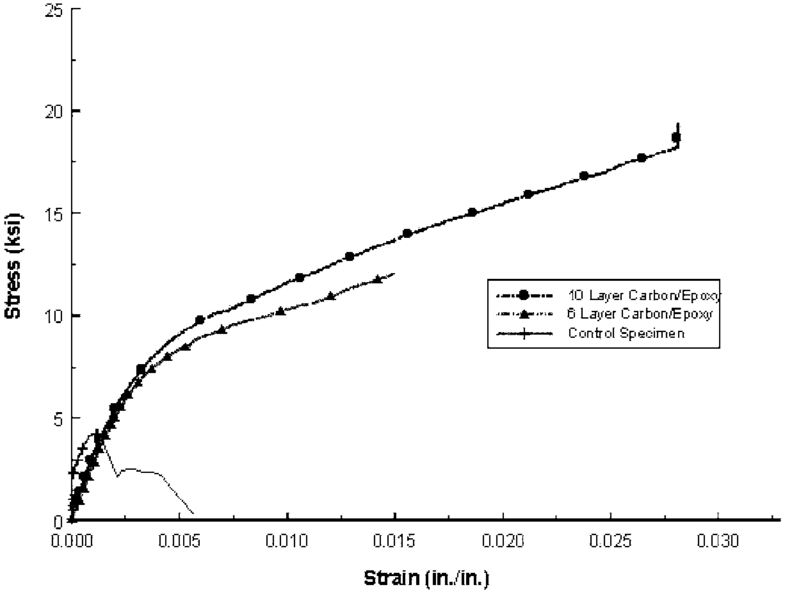


Fig. 31 Experimental stress–strain curves for confined with FRP and unconfined circular columns (Youssef, 2001).

$$f'_{\mathrm{cc}} = f'_{\mathrm{co}}\left[2.25\sqrt{1 + 7.9\,\frac{f'_t}{f'_c}} - 2\,\frac{f'_l}{f'_c} - 1.25\right] \tag{27}$$

where f'_{co} = unconfined compressive strength of the column
f'_{cc} = confined compressive strength of the column
f_l = lateral confining stress = $0.26\rho_{sj} f_{uj} \sin^2\theta$ (28)
$\rho_{sj} = \dfrac{4t_f}{D}$ (29)

Table 4 Rectangular Specimens Summary Test Results

No. of (0) Plies	Unconfined Stress (ksi)	Unconfined Capacity (kips)	Confined Stress (ksi)	Confined Capacity (kips)	Percent Increase in Capacity
		E-Glass/Epoxy Confined Specimens			
3	*4.23*	*634.32*	*5.02*	*753.00*	*18.71*
4	*4.23*	*634.32*	*5.47*	*820.50*	*29.35*
7	*4.23*	*634.32*	*6.24*	*936.00*	*47.56*
11	*4.23*	*634.32*	*7.10*	*1,064.55*	*67.83*
		Carbon/Epoxy Confined Specimens			
2	4.23	634.32	4.81	721.34	13.72
3	4.23	634.32	5.75	862.50	35.97
5	4.23	634.32	6.09	913.50	44.01
8	4.23	634.32	6.30	945.00	48.98

Table 5 Test Results of Hexagonal and Octagonal Column Specimens Confined with FRP Composites

Specimen Shape	No. of (0°) Plies	Unconfined Stress (ksi)	Unconfined Capacity (kips)	Confined Stress (ksi)	Confined Capacity (kips)	Percent Increase in Capacity
		E-Glass/Epoxy Confined Specimens				
Hexagonal	6	3.23	472.73	9.53	1392.61	194.59
Octagonal	6	3.05	485.16	9.85	1566.64	322.91
		Carbon/Epoxy Confined Specimens				
Hexagonal	4	3.23	472.73	8.76	1280.73	170.92
Octagonal	4	3.05	485.16	9.53	1516.92	312.66

f_{uj} = ultimate tensile strength of the composite laminates
t_f = thickness of the composite jacket
D = column's diameter
θ = angle of fiber orientation relative to the member axis $\geq 75°$ (the maximum efficiency for this application is achieved at $\theta = 90°$).

For *a rectangular column,* a similar expression is used as follow:

$$f'_{cc} = f'_c(1 + 1.5\rho_{sj}\cos^2\theta) \tag{30}$$

where

$$\rho_{sj} = 2t_f\,\frac{B + H}{BH} \tag{31}$$

and B, H are the cross-sectional dimensions of the column.

It should be noted that the above equations are based on a confinement model for concrete with steel jackets, which behaves differently than FRP composites. For this reason, several models were proposed to account for the unique properties of composite jackets. One of the early models was proposed by Almusallam and Alsayed (1995). The model proved to be effective in predicting the behavior of concrete axial members confines with FRP composites. We recommend reviewing the development of the current model to account for the linear nature of FRP composites. In 1997, Mirmiran followed the same path and developed a simple model based on small and medium unreinforced circular column specimens. At the present, the use of the Mirmiran model is highly recommended for predicting the strength of "circular" concrete axial members with external composite jackets. This model was developed only for circular concrete columns in which the axial response is bilinear with no descending branch. According to this model, the confined compressive strength of a circular axial member with composite jacket applied in the hoop direction is given by the following equation:

$$f'_{\mathrm{cc}} = f'_{\mathrm{co}} + 4.269 f_l^{(0587)} \tag{32}$$

Figure 32 shows the effectiveness of the Mirmiran model (1997) in predicting the experimental stress–strain curve of a circular standard concrete cylinder 6 in. × 12 in. (152 mm × 304.8 mm).

Additional design information on predicting the ductility enhancement of circular and rectangular columns, and the lap splice confinement gain is described in the ICBO AC125 (ICBO, 2001b) document. The structural engineer is also referred to a useful design textbook by Priestley et al. (1995). As mentioned earlier, the Mirmiran model is only proven to be effective for columns with circular cross sections, and for columns with other geometries, special models are under development at the University of California at Irvine and will be available in the near future.

Durability and Long-Term Performance of Composite Repair Systems

One of the major issues facing the civil engineer when deciding the use of polymer composites in construction applications is durability and long-term performance. For that reason, active programs addressing this subject were initiated by different state and federal agencies, as well as professional organizations such as the U.S. Federal Highway Administration (FHWA), the California Department of Transportation (Caltrans), the International Conference of Building Officials (ICBO), and many other organizations.

Pioneering efforts regarding the durability of FRP composites for infrastructure applications were initiated by Caltrans as a joint project with the Aerospace

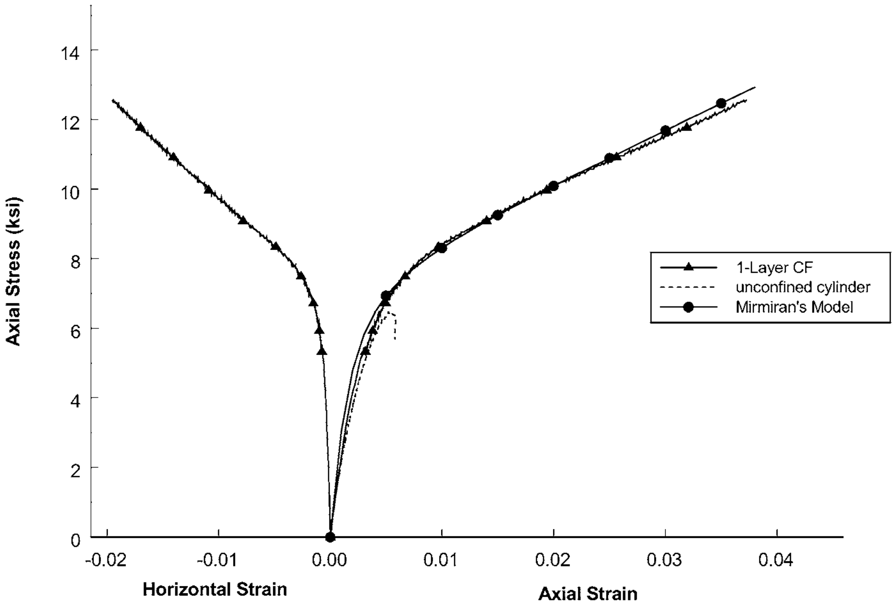


Fig. 32 Successful prediction of the stress–strain behavior of FRP-jacketed unreinforced concrete cylinder using Mirmiran model (Eq. 32).

Corporation (Sultan et al., 1998; Steckel et al., 1999) for composite repair for highway bridge columns. For general building applications, a similar program was developed by the ICBO-ES and is described in details in Tables 1 and 20 of the ICBO AC125 (ICBO, 2001b).

2.3 Internal Reinforcement of Concrete Members Using FRP Composites

FRP composites can also be used as an internal reinforcement for concrete and masonry members. Currently, FRP internal reinforcements are produced in several forms, such as (1) FRP rebars and grid and (2) FRP prestressing cables. There are several applications where composites are the preferred choice as internal reinforcement to concrete and masonry, including:

1. Corrosion environments [e.g., waterfront and marine structures, desalination plants, parking garages and bridges exposed to deicing salts (Fig. 33), and others]
2. Structural members of magnetic resonance imaging (MRI) in hospitals, due to the electromagnetic transparency of composites
3. Electrical applications of E-glass composites internal reinforcement due to its nonconductivity properties that contributes in avoiding electrical-

Fig. 33 FRP composite reinforced bridge deck. (Courtesy of Hughes Brothers Company)

Fig. 34 GFRP and CFRP reinforcing rebars.

related hazards and interference at high-voltage environments (e.g., reinforced concrete power poles, foundations of structural systems in power stations, etc.)

The common form of composite internal reinforcement is FRP rebars made from E-glass (GFRP) and carbon-based composites (CFRP) (Fig. 34). FRP composite rebars are available in standard lengths and diameter grids. According to ACI440 (2001), the tensile strength of commercially produced FRP rebars varies from 70 to 230 ksi (483 to 1600 MPa) for GFRP and from 87 to 535 ksi (600 to 3690 MPa) for CFRP. The longitudinal modulus of elasticity ranges from 5.1 to 7.4 ksi (35 to 51 GPa) for GFRP and from 15.9 to 84 × 10^3 ksi (120 to 580 GPa). The ranges for rupture strain for GFRP and CFRP rebars are 1.2–3.1%, and 0.5–1.7%, respectively. Figure 35 presents a comparison between the mechanical properties of two types of FRP internal reinforcement as compared to conventional steel reinforcing bars.

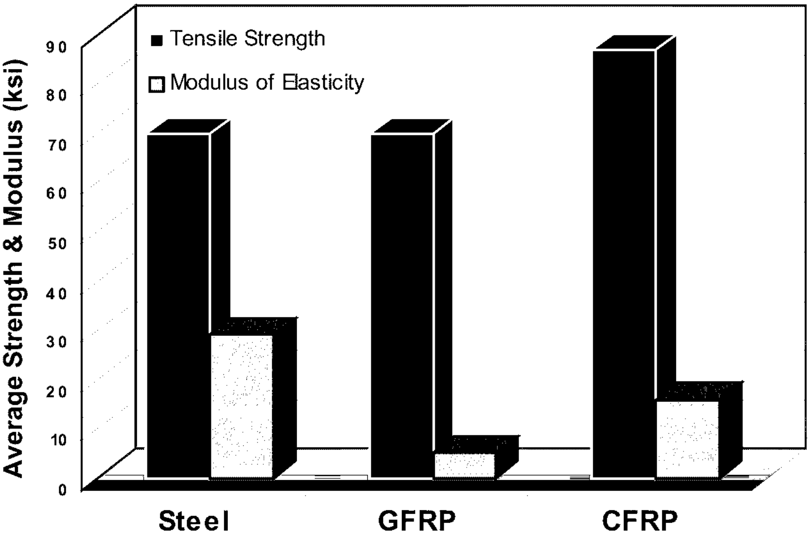


Fig. 35 Comparison between steel and FRP composite rebars mechanical properties (lower bound).

An early study on the use of FRP composites internal reinforcement was initiated by Bank et al. (1991). In this work, a pilot experimental study on the use of FRP grids and gratings as internal reinforcements of one-way concrete slabs was conducted.

The use of GFRP rebars as internal reinforcement for concrete slabs and beams was first initiated in United States at West Virginia University (Faza, 1991). Over the past few years, a number of studies on the durability and long-term performance of FRP internal reinforcement were reported (e.g., GangaRao and Vijay, 1997; Sen et al., 1998; Porter et al., 1995, Alsayed et al., 2001). The majority of the durability studies concluded the sensitivity of GFRP reinforcing materials to alkaline environment found in fresh concrete. The strength degradation of GFRP rebars can reach values up to 75%, while the stiffness degradation, in many cases, can reach to a value up to 20% (ACI440, 2001).

For this reason, it is the author's recommendation to limit the use of GFRP as primary reinforcement in a high pH alkaline environment to low stress level exposure to minimize the possibility of the development of microcracks in the matrix, which opens the doors for alkaline attack of the E-glass. Another alternative is using alkaline-resistant (AR) glass fibers, although the cost may be higher relative to E-glass fibers. For heavier stress environments, carbon-based composite reinforcements are highly recommended. Again the cost may be the issue, but the reliability in this particular environment is higher.

For a comprehensive coverage of the construction and design aspects of FRP composite internal reinforcement of concrete members, the reader is referred to a recent document published by the American Concrete Institute (ACI440.1R-01, 2001).

2.4 All-Composites Structural Applications

In addition to the repair and reinforcement application of composites in construction, composite materials are being used to build the entire structure such as warehouses, buildings, highway bridge decks, and other civil engineering structures. One of the popular types of composites in construction applications is pultruded composites. For decades, pultruded fiber-reinforced polymeric (PFRP) composites have been used as secondary structural members in several construction applications such as petrochemical plants plate forms, cooling tower structures, and in water and wastewater treatment plants applications. The pultrusion process is a continuous manufacturing process where the saturated fibers are pulled through heated die using continuous pulling equipment. The hardening or gelation of the resin is initiated by the heat from the die producing a cured rigid pultruded profile that is cut to length by an automated saw (refer to Fig. 36). Pultrusion is considered to be the only closed-mold process that allows for combining a variety of reinforcement types and hybrids in the same section. Most of the commercially produced PFRP structural shapes are composed of multilayers of surfacing veil or Nexus, continuous fibers (roving), and continuous strand mat. The typical volume fraction of fibers for "off-the-shelf" sections is in the range of 40–45%. A variety of structural profiles (open and closed web) are now available similar to steel sections (H, I, C, L, . . .). The major reinforcements of these sections are concentrated in the longitudinal direction of the section with minimum reinforcement in the transverse direction. The most

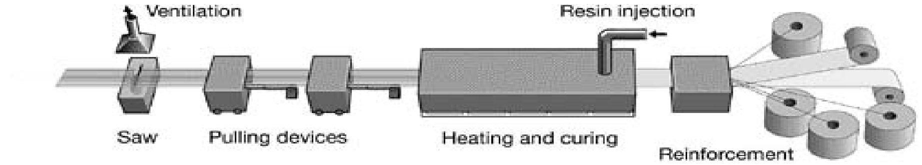


Fig. 36 Pultrusion process. (Courtesy of Fiberline Composites A/S)

common fiber type is the E-glass in the form of rovings and strand mats. However, recently, carbon/E-glass composite profiles have been produced in limited bridge applications.

As shown in Fig. 37, with few exceptions, the majority of the off-the-shelf pultruded profiles is similar, in geometry, to steel profiles and are commercially available in different sizes and grades [Fiberline (2000), Bedford (1999), Creative Pultrusions (1985), Strongwell (1990)].

Although the use of unidirectional reinforcement schedule may be satisfactory for lightweight or secondary structural members, it indeed is not sufficient for primary structural carrying members such as bridge decks, girders, and columns. Other disadvantages of using thin-walled unidirectional "steel-like" PFRP profiles include the insufficient lateral and buckling resistance of the section. In addition, in the majority of commercially produced unidirectional open web (e.g., H-profile, channels, angles, etc.) and closed-web (e.g. rectangular and box profiles) there is a lack of fiber continuity between the web(s) and flanges. For this reason a premature failure at the web–flange junction is the common mode of failure of such profiles. A comprehensive discussion on this issue is reported by Mosallam (1993, 1996).

Research and Development of PFRP Composite Structures

In the late 1980s, several major research projects were initiated to study the structural performance of pultruded composite structures. In 1990, Mosallam conducted a comprehensive study on the behavior of PFRP portal frame. The

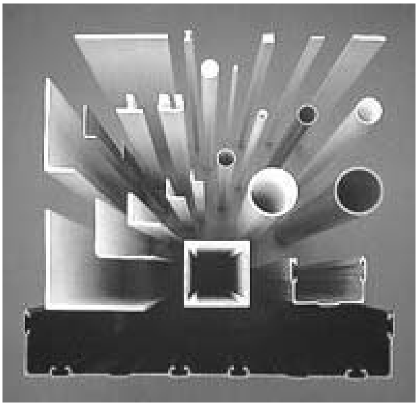


Fig. 37 Sample of pultruded composites profiles. (Courtesy of Fiberline Composites A/S)

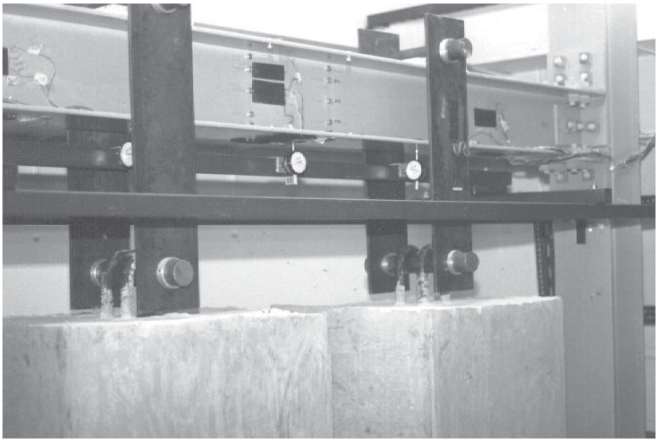


Fig. 38 10,000-h full-scale creep test of a PFRP composite portal frame (Mosallam, 1990).

study included both full-scale experimental testing and theoretical modeling. The experimental part focused at the creep behavior (Fig. 38), service and ultimate behavior, framing connections, and buckling and postbuckling performance of PFRP frames. Simple expressions for the viscoelastic moduli (axial and shear) of the PFRP composites were developed. The ultimate mode of failure and the effect of the nonlinearity of the framing joints on the stiffness and buckling behavior of the pultruded thin-walled sections were also performed (Bank and Mosallam, 1992). Based on the test results (Mosallam, 1990), a premature local failure of open-web unidirectional PFRP sections occurred due to the inadequacy of reinforcement continuity at the web–flange junction (Fig. 39). This premature failure caused by the separation of the web and the flanges of the open-web PFRP elements at the stress concentration locations (usually at the connections and the girder midspan) affects the general behavior of the structure. For ex-

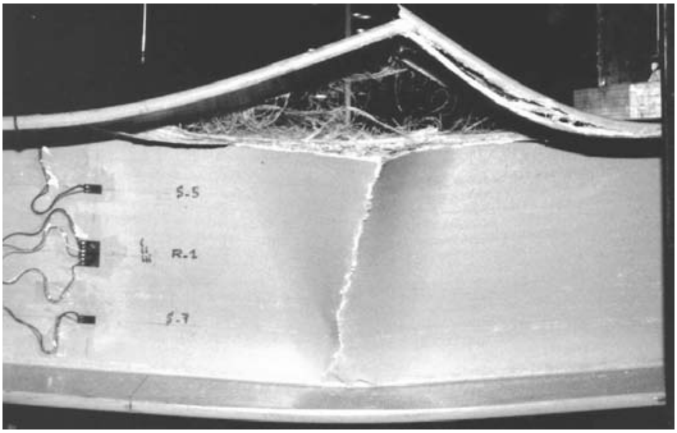


Fig. 39 Failure of web/flange junction of PFRP open-web profiles (Mosallam, 1990).

ample, results of experimental and theoretical research work (Mosallam, 1993; Mosallam et al., 1993) showed the direct effect of this premature failure of the column section of frame structures in the rotational stiffness PFRP frame connections during the crack growth up to the failure. The loss of the flexural stiffness results in a decrease of the connection rotational capacity, and consequently an increase in the flexural stresses at the girder midspan of a PFRP frame structure. Simple reinforcement techniques for overcoming this pre-mature failure were reported by Mosallam (1996).

Unlike aerospace-type joints where the majority of applications is concerned with lap splice joints, the majority of composite joints used in construction applications involve frame connections.

For any frame structure, connections are considered to be of the most critical structural elements, which play a major role in controlling both the serviceability and ultimate strength of the PFRP frame structures. Careful design of the connecting elements will ensure both the safety and the efficient use of the material. Previous studies on PFRP frame structures (Mosallam, 1990; Bank et al., 1990) showed that a premature failure of pultruded shapes would occur if a wrong connection details were used (refer to Fig. 40). Based on this fact, Bank et al. (1992) have extended this work by introducing different connection details to overcome the premature failure of the pultruded shapes at the web–flange junction of PFRP H-beams. The connection details presented in their study considered the anisotropic properties of the PFRP structures. Their results showed that maximum strength and maximum stiffness could be achieved by using a connection with both mechanical and adhesive elements.

All PFRP connections developed and tested in all previous studies (e.g., Bank et al., 1992) have utilized PFRP connecting elements, which were commercially produced and were not intended specifically for connecting purposes. This was an appropriate approach to demonstrate the deficiency of the existing connection details, as well as to provide strengthening details for reinforcing the existing PFRP connections. To overcome this problem, a different approach for connecting PFRP structural elements to ensure the prosperity and the efficient and

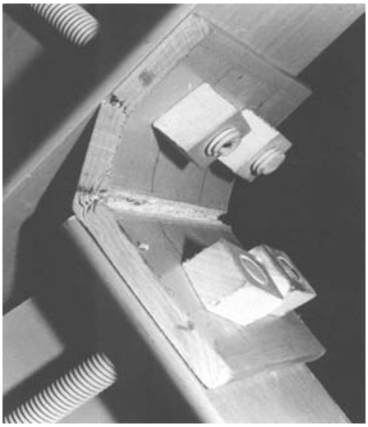


Fig. 40 Premature failure of steellike connection details (Mosallam, 1993).

safe use of this material should be used. This approach is to develop a special connecting element or system using a mixture of past experience, available research and design data, and knowledge of the anisotropic behavior of the composite materials. The design criteria of the connecting elements include proper fiber orientation, ease of erection and duplication, geometrical flexibility of the use for different structural connections, and maximizing both the overall connection stiffness and ultimate capacity. Based on these criteria, a custom-made FRP prototype connector was developed and was fabricated [using resin transfer molding (RTM)] from E-glass/vinylester composition. This FRP-connecting element [designated, herein, as the universal connector (UC)] was developed by Mosallam (1993). The UC element can be used for the majority of PFRP connection details for joining different structural shapes, e.g., exterior and interior beam-to-column connection, column–base connections, continuous beam connections, beam-to-girder connections, and others (Fig. 41). An extensive theoretical and experimental program on the development and characterization of PFRP connections is in progress.

The dynamic response of both PFRP materials and structures was investigated by Mosallam et al. (1993). In this study, results of experimental dynamic tests of FRP pultruded structural elements and framed structures were presented. The thin-walled elements used in this study were standard "off-the-shelf" pultruded 4 in. (101.6 mm) × 4 in. (101.6 mm) × $\frac{1}{4}$ in. (6.35 mm) H-beam and 2 in. (50.8 mm) × 2 in. (50.8 mm) × $\frac{1}{4}$ in. (6.35 mm) square tube made of E-glass–polyester composition. All the connectors and connection elements were made of PFRP threaded rods, nuts, and high-strength epoxy adhesive. The test specimens in this study were excited dynamically using both impact loading and shaking loads. Experimental modal analysis was used to extract the natural frequencies, modal damping, and mode shapes of the test specimens. Comparison between two types of frame connections was also performed to determine the effect of using high-strength adhesives. The study further showed the validity of using both the material properties and the lay-up of the coupons in modeling PFRP beams and frame structures.

A pilot study on evaluating the structural cyclic performance of composite frame connections for pultruded structural systems was conducted by Mosallam (1999). In this study, several full-scale cyclic tests were conducted on several

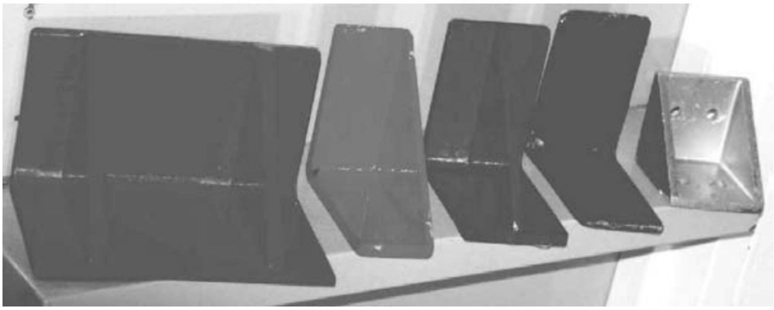


Fig. 41 FRP composite continuous universal connectors for PFRP framing joints. (Courtesy of Sigma Composites, LLC)

pultruded framing elements (Fig. 42). This included box and H-beam profiles with different sizes. The emphasis of this study was on interior framing connections with both flange and web attachments. In addition to high-strength adhesives, both FRP and steel mechanical fasteners were studied. Bolted-only, adhesively bonded only, and combined joint details were evaluated using both metallic and nonmetallic bolts. Strain, deflection, and load information were collected using a computerized data acquisition system. Hysteresis curves M/θ and P/δ were developed and analyzed (Fig. 43). For FRP mechanical fasteners bolted-only connections, a common mode of failure was observed for all specimens. This was a combination of bolt thread shaving and flexural fatigue-type failure of pultruded threaded rods. Other local failures to the pultruded thin-walled beam sections were observed at the ultimate moment. Delamination cohesive failures were also observed for adhesively bonded connection details.

Currently, a pilot experimental program has been initiated by the author on the seismic behavior of PFRP three-dimensional frame structures (Fig. 44). In this program, both one- and two-story three-dimensional frames made entirely from PFRP composites and gratings are evaluated under ground motion. The tests focus on evaluating the effect of different connection details on the dynamic response of the PFRP frame structure.

Construction Applications of PFRP Composites

Buildings Applications. Several projects have been constructed entirely using pultruded fiber-reinforced polymer (PFRP) composite sections as the main structural elements. One of the early applications is the construction of four PFRP turret towers on top of the Sun Bank Building, Orlando, Florida. Figure 45 shows the framing of one of the three-story high towers, which was built entirely from PFRP shapes (H, angles, threaded rods, and nuts). All columns and girders were constructed using open-web H sections, which were connected together using FRP bolts and nuts. The use of PFRP composites was the preferred choice because of the electromagnetic transparency and radio wave reflection properties of composites. Due to the nonmagnetic properties of PFRP

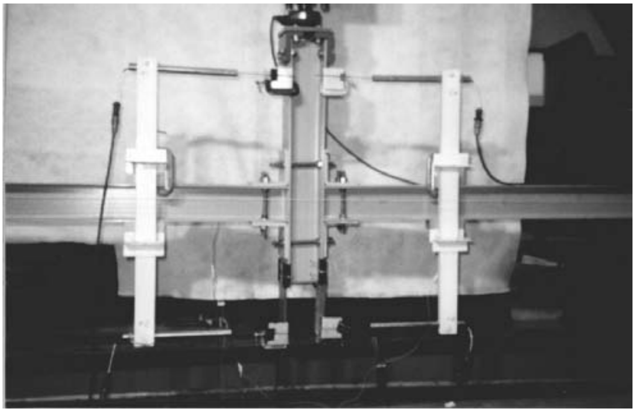


Fig. 42 Cyclic behavior of PFRP composite frame joints (Mosallam, 1999).

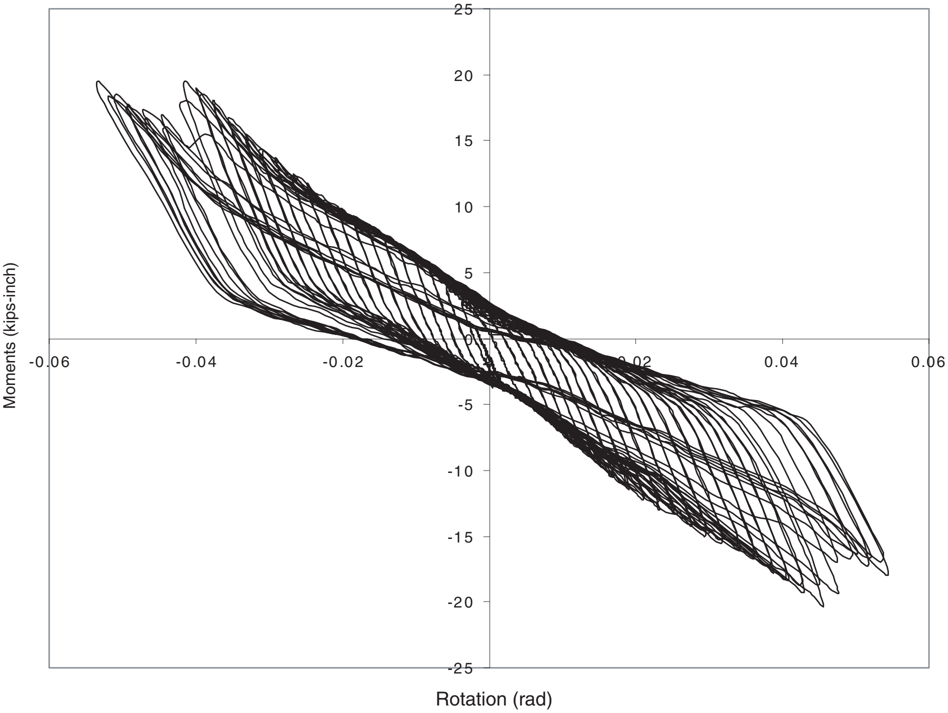


Fig. 43 Typical M/Θ hysteresis of PFRP composite frame joint (Mosallam, 1999).

Fig. 44 Seismic evaluation of three-dimensional PFRP frame structure with PFRP gratings.

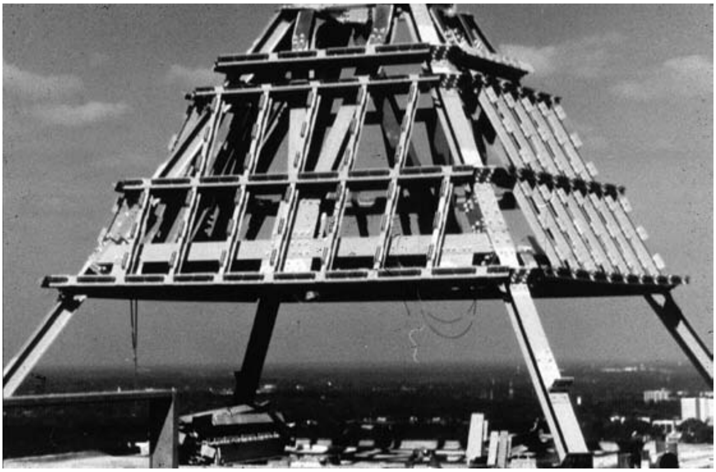


Fig. 45 Three-story high towers framing of the Sun Bank Building, Orlando, Florida. (Courtesy of Strongwell Company)

composites, it is commonly used for facilities with delicate instrumentation. Figures 42 and 44 show a complete frame structure, which was constructed using PFRP materials. The ease of fabrication, transportation, and erection resulted in shorter construction time.

The first residential/office building with PFRP structural profiles was presented as the *Eyecatcher Project* at the Swissbau'99 Fair in Basel, Switzerland. After the exhibition, the construction was disassembled and brought to its new location at Münchensteinerstrasse 210, Basel, where it now serves as a permanent office building. The Eyecatcher all-composite building is open to the public on agreement. The height of the all-composite 5-story building is 15 m (49.21 ft) (with a ground-floor area of 10 × 12 m (30.48 × 39.37 ft). The inclined and vertical columns were fabricated as a buildup section made of one H-profile and two U-profiles. The horizontal frame girders were also built-up sections made of two U-profiles and four flat pultruded plates. In all builtup sections, the pultruded composite profiles were bonded using high-strength epoxy and were subsequently bolted together with steel bolts. Figure 46 shows the skeletons during construction and the finished office building. Other examples of composite framing structures are shown in Figs. 47 and 48.

Bridge Applications. In the United States, there are over 90,000 weight-restricted bridges. In most cases, there are no funds allocated to solve the problem by replacing these decks. These bridges are frequently replaced with a modern multigirder design to restore the route to traffic without weight restrictions. To replace the bridge would have cost $2.4 million. In the past few years, FRP composite decks have proven to be an ideal solution to this problem, with a cost reduction of up to 30% as well as the tremendous saving in construction time and traffic interruption.

In the past few years, the U.S. Department to Transportation (DOT) has utilized composite decks to replace corroded and underrated bridge decks. For ex-

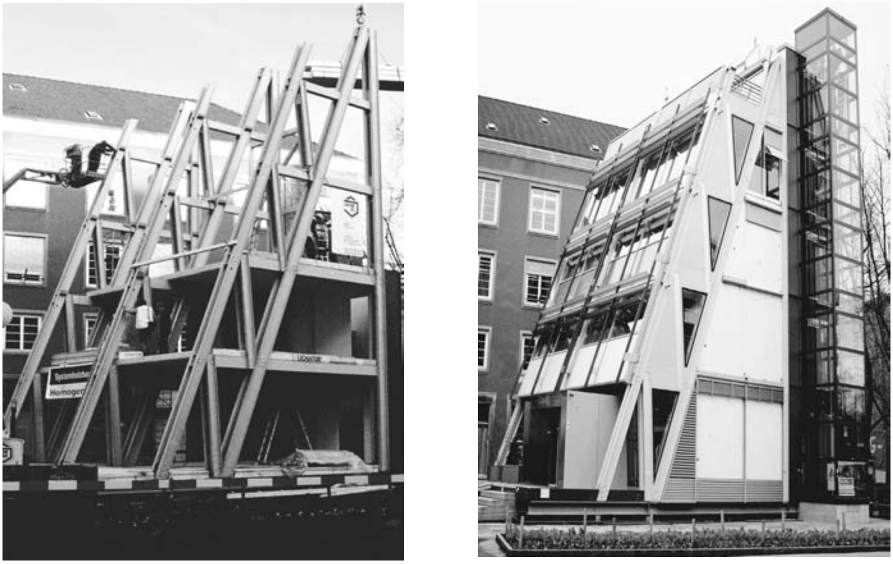


Fig. 46 All-composite ″Eyecatcher″ building in Switzerland: (a) pultruded frame skeletons and (b) the completed structure. (Courtesy of Fiberline Composites A/S)

Fig. 47 Pultruded composites frame structure. (Courtesy of Strongwell Company)

Fig. 48 All-composites skeleton. (Courtesy of Strongwell Company)

ample, the New York DOT has selected the composite deck solution to replace the old deck of the Chemung County Bridge. This steel truss bridge was originally built in 1940, with a span length of 140 ft (42.7 m) and a width 24 ft (7.32 m). The average daily traffic on this bridge (AADT) is 3250 and 7% of this volume is trucks. There were several factors contributing to the posting problem of this bridge, which is typical of the majority of old steel bridges, including:

- Over the past 60 years, little more than a new course of asphalt every few years was added to smooth the wearing surface. This resulted in an increase of dead load.
- In addition, some steel sections were rusted.
- In the original design, the bridge was never intended to carry the heavy loads on the road today.

The engineers in New York DOT Region 6 decided to adopt the FRP composite lightweight solution (refer to Fig. (49), in addition to repairing and painting the steel truss members. After the addition of the FRP bridge deck, the load rating of the bridge raises from the original *inventory* of HS12 (22 tons) with *operating* capacity of HS18 (33 tons) to an inventory of HS23 (42 tons) with operating capacity of HS34 (61 tons). Figures 50 and 51 show the FRP bridge deck during fabrication and installation, respectively.

Another major highway bridge project that utilized FRP composite decks is the Salem Avenue Bridge located just west of downtown Dayton, Ohio. Four different FRP composite decks were used.

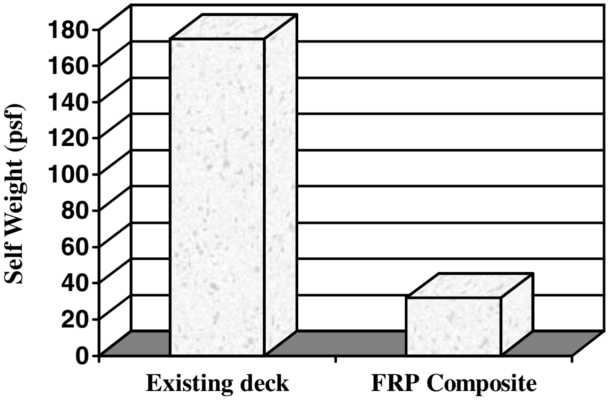


Fig. 49 Self-weight comparison between existing and new FRP composite deck of the Bentley Creek, Chemung County, New York.

1. *CDS Deck.* Consists of FRP stay-in-place forms that act as bottom reinforcement for the composite/FRP hybrid deck. The top layer of reinforcement consists of GFRP rebars. The composite forms were filled with high-strength concrete.

2. *CPI Deck.* Consists of pultruded interlocking profiles that run transverse to the bridge centerline and are adhesively bonded at the shop to form 8-ft-wide (2.43-m) composite panels (refer to Fig. 52). The panels were attached to the existing steel girders using Nelson shear studs that were welded to the top of the steel girders.

3. *HCI Deck.* Coinsists of a sandwich panel with high-density foam core that were manufactured using Seemman Composite Resin Infusion Molding Process (SCRIMP). The composite panels were connected to the steel girders using Nelson studs.

4. *ICI Deck.* Consists of deep sandwich panels with prefabricated corrugated and straight E-glass composite shells bonded together. Same connectors were used.

Fig. 50 Fabrication of the FRP composite bridge deck of NY 367 over Bentley Creek, Chemung County, New York. (Courtesy of New York DOT, Region 6, Hornell, NY)

Fig. 51 Installation of all-composite bridge deck of NY 367 over Bentley Creek, Chemung County, New York (2000). (Courtesy of New York DOT, Region 6, Hornell, NY)

The latest application of all-composite deck for a highway steel bridge has been initiated by Caltrans. A hybrid (carbon/E-glass) sandwich composite deck developed by Martin Marietta (Fig. 53) is planned to be installed at the Schuyler Heim steel lift bridge in Long Beach, California. The 1212 ft (370 m) long, four-lane bridge provides vehicles access to Terminal Island in the Port of Long Beach. The middle lift portion of this bridge uses a 224-ft, (68.3-m) lightweight open-grated steel deck. Due to the high volume of heavy trucks crossing the bridge in the recent year, the welded steel deck suffered from a chronic fatigue problem, and Caltrans was forced to replace portions of the steel deck periodically. Due to the excellent fatigue performance of composites, the preferred choice was replacing the existing steel deck with all-composite hybrid deck system (Hranac, 2001). Small- and full-scale tests were conducted on several

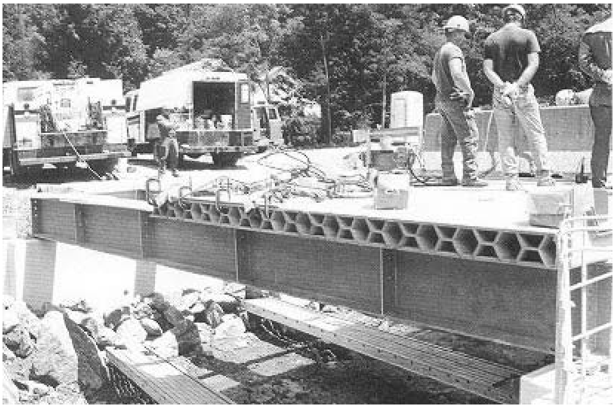


Fig. 52 Pultruded Interlocking composite deck developed by West Virginia University (GangaRao, 2000).

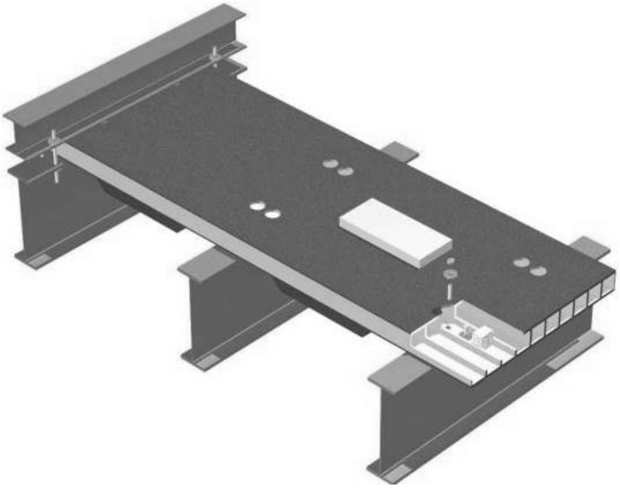
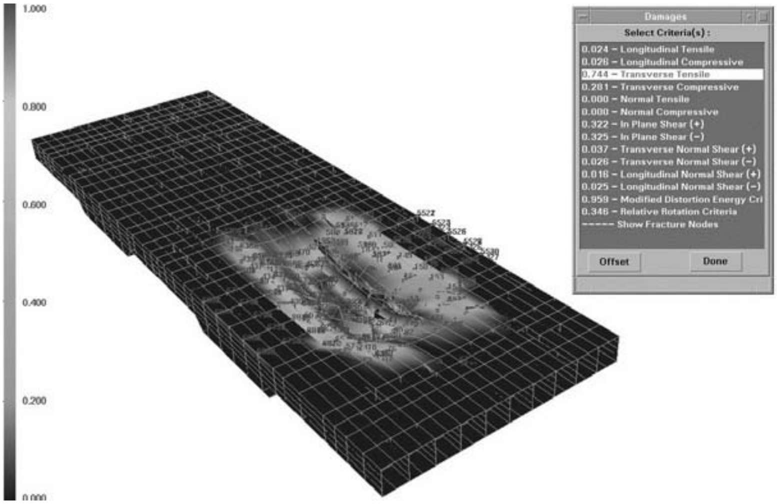


Fig. 53 All-composite hybrid deck system for the Schuyler Heim Bridge, Long Beach, California. (Courtesy of Martin Marietta Composites)

composite panels at both California State University at Fullerton and University of California at Irvine (refer to Fig. 54). Test results indicated that the FRP composite deck has exceeded both the strength and stiffness criteria required by the DOT. The composite deck will be instrumented with different measuring devices and will be monitored for a period of approximately one year. Details of the structural evaluation are reported by Mosallam and Haroun (2001). The destructive test was simulated using a state-of-the-art virtual testing/progressive analysis software called GENOA (Alpha Star, 2001) as shown in Fig. 55.

In Europe, several all-composite bridges and bridge decks have been built. The world's first all-composite cable-stayed footbridge (called the Aberteldy Bridge) was constructed in Scotland and opened on June 2, 1992. The Aberteldy

Fig. 54 Full-scale destructive tests on all-composite hybrid deck system for the Schuyler Heim Bridge, Long Beach, California. (Courtesy of University of California, Irvine, and California State University, Fullerton)



0.024 – Longitudinal Tensile
0.026 – Longitudinal Compressive
0.744 – Transverse Tensile
0.281 – Transverse Compressive
0.000 – Normal Tensile
0.000 – Normal Compressive
0.322 – In Plane Shear (+)
0.325 – In Plane Shear (–)
0.037 – Transverse Normal Shear (+)
0.026 – Transverse Normal Shear (–)
0.018 – Longitudinal Normal Shear (+)
0.025 – Longitudinal Normal Shear (–)
0.959 – Modified Distortion Energy Cri
0.346 – Relative Rotation Criteria
------ Show Fracture Nodes

Fig. 55 Full-scale simulated destructive test using GENOA Progressive failure program. (Courtesy of Alpha Star Corporation, Long Beach, California)

Bridge is 2.2 m (7.2 ft) in width and total span of 113 m (370 ft), with a mainspan of 63 m (206 ft) between the two A-frame composite pylons, as shown in Fig. 56. The height of each composite A-frame pylon that support the mainspan with Kevlar composite fan-type cables is 17.20 m (56.43 ft). The structural components of the bridge deck, A-frame pylons, and handrails were all made of GFRP, while the cable stays were made of dry parallel aramid fibers (Kevlar 49) in polyethylene sheaths. The total cost of the bridge was about $200,000 (£120,000), in addition to some donated labor. After nearly 10 years of service, slight sagging of the bridge occurred that can be attributed to the viscoelastic nature and the low modulus of aramid cables. Table 6 presents the physical and

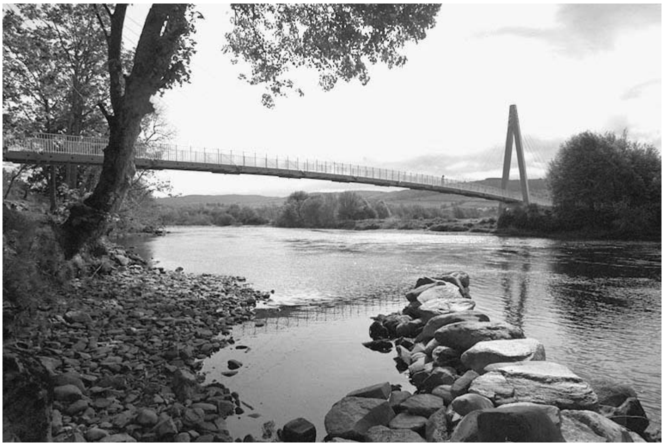


Fig. 56 Aberteldy all-composites cable-stay bridge. (Courtesy of Strongwell Company)

Table 6 Physical and Mechanical Properties of the Different Composite Components of the Aberfeldy Bridge

Composite Materials and Properties	Deck/Pylons	Cable Stays
Fibers	E glass	(Kevlar® 49)
Matrix	Isophthalic polyester resin	None
Manufacturing process	Pultrusion	Parallel Sheathing
Assembly	Toggle and epoxy bonding	—
Adhesives	Epoxy	—
Tensile modulus	22 GPa (3.2 × 10^6 psi)	127 GPa (18.4 × 10^6 psi)
Tensile strength	300 MPa (4.3 ksi)	1.9 GPa (276 ksi)
Ultimate tensile strain	1.4%	1.6%

From FHWA (1997).

the mechanical properties of the different composite components of the Aberfeldy Bridge (FHWA, 1997).

A similar all-PFRP-composite pedestrian cable-stayed bridge was built by Fiberline Company (Kolding, Denmark) crossing a busy rail line and was officially opened on June 18, 1997 (Fig. 57). Although the construction work was restricted to only a few hours during weekend nights due to the busy railway line, restricted installation work to only the bridge was fully installed in only three short nights. The short installation time has illustrated the clear advantages of composites.

In 1997, an all-composite bridge was installed in the mountainous region of Pontresina in Switzerland (refer to Fig. 58). The two bridge sections, each measuring 12.5 m (41 ft) and weighing a total of 2,500 kg, were placed by a helicopter, one section at a time. The load-carrying capacity of this bridge is 500 kg/m^2, in addition to a 1-ton snow-clearing vehicle allowance.

Fig. 57 Fiberline all-composite cable-stayed in Denmark. (Courtesy of Fiberline Composites A/S)

Fig. 58 Pontresina all-composite truss bridge constructed in Switzerland in 1997. (Courtesy of Fiberline Composites A/S)

Currently, a new generation of optimized pultruded profiles for a modular bridge deck is being developed through the ASSET project supported by the European Union. The pultruded sections are being manufactured by Fiberline Composites A/S of Denmark. This bridge is considered the first highway bridge that has a load capacity up to 40 tons and will be constructed across a motorway in Oxfordshire in England during the summer of 2002. Figure 59 shows one of the profiles that will be used in this composite bridge deck. A comprehensive review of other composite bridges can be found in the FHWA report (1997).

3 DEVELOPMENT OF CODES AND STANDARDS

In recent years, the construction industry started to realize the potential of using polymer composites in construction applications. Unfortunately, the construction industry and the civil engineers were faced with a tremendous amount of difficulties to utilize these materials in the same manner they are used to for the

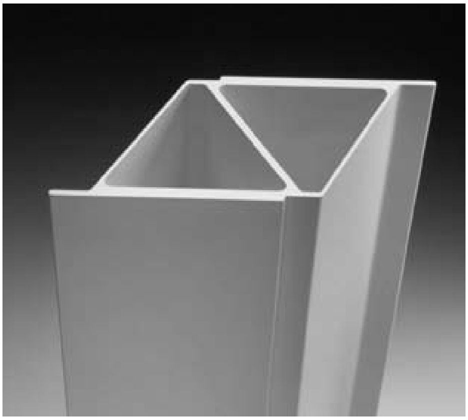


Fig. 59 Optimized patented pultruded profile for modular composite highway bridge deck applications. (Courtesy of Fiberline Composites A/S)

conventional material such as steel, concrete, and wood. The major obstacle is the lack of design standards and authoritative codes for the use of these materials in construction applications. Despite the fact that there is a great deal of research and application information available from the aerospace industry for the past four decades or so, still the civil engineers are searching for ways to convince them with the reliability, applicability, and the structural efficiency of such materials. The Structural Composites and Plastics Committee (SCAP) of the American Society of Civil Engineers (ASCE) appreciates this demand and is working to assist the civil engineers to achieve this goal.

For any structural system, design standards are one the essential requirements for professional engineers' acceptance. Both the American Concrete Institute (ACI) and the American Society of Civil Engineers (ASCE) has been involved in the development of several standard documents for different materials and systems. Since the 1960s, the ASCE has been involved in developing several engineering documents dealing with both unreinforced and fiber-reinforced polymers (FRP) materials and systems. In 1984, the ASCE Structural Plastics Design Manual (SPDM) was published (1984) by the Plastic Research Council of the Materials Division of ASCE. Starting in the late 1980s, as the demand and the acceptance of FRP materials increased, the ASCE recognized the need for more developments in this field. Jointly with the Society of Plastics Industry (SPI), a long-range, multiphase program was established in the early 1990s. The ultimate goal of this joint program is to develop accepted standards for structural design, fabrication, and erection of FRP composite systems. In 1995, the Pultrusion Industry Council (PIC) of SPI sponsored the first phase of this program to develop a design draft standard or a "prestandard" document with a view to process the prestandard upon completion as an ASCE national consensus standard in accordance with the rules of the American National Standard Institute (ANSI).

In late 1995, ASCE awarded phase I of this project to Chambers Engineering, p.c. (as the general contractor) and the author (as the subcontractor) to undertake a one-year startup and planning phase of the multiphase standard program. The scope of work of phase I was to (i) surveying and evaluating existing design and martial information. This task included researching both published and unpublished technical literature, government and university reports, performance data, standards and specification documents (ASTM, ACI, ASCE, JSCE, Eurocode, Canada), manufacturer's materials data, and current practice relative to the use of FRP composites. (ii) Development of a computerized database containing the relevant and evaluated useful technical information, (iii) using this database, identify gaps in knowledge that might impede promulgation of the standard, and (iv) developing the prestandard outline through defining the approach including recommended design philosophy and relationship of the ASCE design standard with other martial or industry standards such as AASHTO, ASTM, ISO, ICBO, and other test standards.

In 1993, the American Concrete Institute realized the potential of the polymer composites in concrete applications. For that reason, a new committee (ACI440) was formed to answer the needs of this new industry and to provide guidelines for design, specifications, and applications of polymer composites as external and internal reinforcement systems. Due to the rapid increase of new polymer composite products and applications, the ACI440 committee was divided into several subcommittees focusing on different design and application aspects of polymer composites in concrete applications. This includes subcommittees on internal reinforcements (FRP rebars), FRP prestressing, FRP external repair, education, and others. One of the active subcommittees is the ACI440 subcommittee on FRP external reinforcements (ACI440F). The ACI440 committee is currently in the final development phase of a design and construction of externally bonded FRP systems for strengthening concrete structures document that will be available shortly to the public. The information presented in the document will assist the structural engineer in properly selecting and designing an optimum and reliable FRP system. The documents also describe conditions where FRP strengthening is beneficial and where its use may be limited.

In 1997, the International Conference for Building Officials (ICBO) evaluation services produced acceptance criteria (AC125) for seismic repair and rehabilitation of reinforced concrete members and walls. Unlike the ACI current proposed document, AC125 focused more on applications related to seismic design. However, the document has been reviewed recently by the different experts and some modifications have been suggested and will be incorporated in the near future.

Lately, the author has developed the *ASCE Structural Design Manual on Pultruded Composite Joints* and will be available in 2002. The manual consists of 12 chapters covering a wide range of design topics related to joining PFRP frame structures (Mosallam, 2001).

4 NEW STRATEGY AND RECOMMENDATIONS

One of the major questions that need to be answered clearly for the structural engineer who has been dealing for decades with conventional materials such as steel and concrete is: Why should he or she select this new material? The same question was asked 40 years ago when composites were first introduced to the aerospace industry. The answer of this question to the civil engineering community is not as simple as many engineers think. The complexity lays on the apparent burden that this engineer will face and the sacrifices expected by dealing with more advanced design and relatively complex structural material. Some of these fears are: (i) the absence of authoritative codes and material specifications, (ii) the lack of simplistic design procedures similar to those that have been established for centuries for conventional materials (concrete, steel, wood), (iii) the direct involvement of the structural engineer in the manufacturing and the tailoring of the material, (iv) the lack of long-term structural and environmental test data, and lastly (v) the need of relatively skilled labors at both the fabrication and construction sites, as compared with composite materials. The effective tool to overcome these roadblocks, in the author's opinion, is education. First, the construction industry should be educated about the nature of these materials and the associated benefits, as well as the special mechanical properties of PFRP composites such as the anisotropy and the viscoelasticity. Second, a modified or new civil engineering curriculum should be established that includes several composite design courses similar to those for steel, wood, and concrete. Generation of structural engineers equipped with the skills required for adopting advanced composites. This, of course, requires the establishment of a design code and specifications. Recently, positive movements by the different engineering and industrial organizations have been initiated to pursue this important task. For example, a new subcommittee of the ASCE plastics and structural composites has been formed to initiate an initial proposal for establishing an ASCE code for composites. The Pultrusion Industry Council (PIC) of the Society of Plastic Industry has already initiated the first phase of a structural design manual project.

It is also important to identify several facts for the civil engineer who will be introduced to FRP composites for the first time. First, the fact that he or she for decades has been using some form of composites with no difficulties (e.g., reinforced concrete, natural, and laminated wood). Second, the engineer should understand that the aim of using FRP composites is neither to replace nor to compete with other construction industries such as concrete, steel, wood, etc. In contrast, these materials are here to enhance and assist the conventional materials in certain applications beyond their capabilities. In addition, these materials if used properly with other conventional materials can produce an optimum engineered structural ‘‘system’’ capable of solving many problems associated with our infrastructures. This misconception has led to both confusion and defensive position of loyal concrete, steel, and wood users. The fact is composites are the right choice in some applications when other construction materials are disqualified in part or whole. For this reason, the structural engineer should begin to look at different materials as elements of an ‘‘integrated structural system.’’

From the above discussion, a new strategy to deal with advanced construction composites is needed. Composites should not be penalizing by adopting the wrong design of sections and structures. It is important for those engineers who are willing to take advantages of these materials to learn the basic mechanics of composites. The directionally dependent, the viscoelasticity, and environmental properties need to be clearly understood. The composite industry must offer all the support and encouragement to all academic and research studies. The great attention shown lately by the different professional and federal organizations in promoting and supporting research and demonstration projects is essential. For example, the recent activities conducted by the Civil Engineering Research Foundation (CERF), the American Society of Civil Engineers (ASCE) SCAP Committee, the International Conference of Building Officials (ICBO), The American Concrete Institute (ACI), the U.S. Corps of Engineers, the Federal Highway Administration (FHWA) Transportation Equity Act (TEA-21) (Hooks, 2000), and the National Science Foundation (NSF) indicate the acceptability and the high level of awareness by the engineering communities. The need of building design codes and material specifications is a must in order for this material to enter and to survive the complexity of the construction industry. Much research data has been conducted in this area that need to be collected in a database system. The importance of this database is to evaluate these works by committee experts in this area to select the useful information for the development of unified design procedures. Experimental and analytical research work on the long-term performance of PFRP is needed to increase the confidence level of these materials. Composite education at the university level and the practical training programs for structural engineers are essential.

As we entered the twenty-first century with these advanced materials, we need advanced design tools to achieve the optimum design and the best performance of these materials. For the other conventional construction materials, the use of simplified design formulas, charts, and tables were acceptable. Fortunately, and due to revolutionary advancement in computer and computational techniques such as finite-element method (FEM), the use of computers will be the recommended tool for composites. This is due to the fact that the optimum design and performance prediction of campsites can only be achieved with more detailed calculations. Simplified equations are still important for quick estimate and as a tool for comparison. In short, advanced composites provide the structural engineers, for the first time, with the challenging opportunity to create innovative structural systems.

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CHAPTER 46

DESIGN FOR MANUFACTURE AND ASSEMBLY WITH PLASTICS

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1 INTRODUCTION

To write a chapter in a book such as the *Handbook of Materials Selection* you need a good introduction or an opening that sets the pace of the information one wishes to present.

From the title we will only be dealing with plastic materials used for designed parts; however, some of the hints, tips, criteria, and suggestions may apply to other materials such as metals and ceramics.

I would have liked to start this chapter with an excellent illustration of a plastic part design. But the best I could come up with is a metal example and hopefully throughout this chapter I will refer to some plastic designs.

The example I would have liked to use is the long swords used by King Arthur and the Round Table. From a distance one can hardly imagine the breadth of one who uses such a long sword in battle and who swings this enormous sword at an enemy to chop off various body parts. The sword was hollow and partially filled with mercury. The sword was relatively easy to manipulate in the so-called rest or nonbattle position. When the knight raised the sword, the mercury being a liquid flowed to the handle. As the sword was swung, the mercury flowed to the tip of the sword, thus giving it extra force at the tip. In my opinion, this is a brilliant example of materials selection in the development of a product. Imagine having to lift the sword if it were solid.

Handbook of Materials Selection. Edited by Myer Kutz

This chapter will consider three main topics: plastic materials selection, plastics joining techniques, and plastic part design. The major focus will be on plastic materials selection. The information in this chapter will be based upon both the lectures I gave and the information I received in short courses taught to practicing engineers and scientists that were involved in all the aspects of commercial plastics part designs and in graduate school courses to budding new materials scientists and engineers.

If you search the open literature for material selection, you will find articles with titles similar to ‘‘The Science of Material Selection’’ or the ‘‘Art of Material Selection.’’ Hopefully this chapter will eliminate some of the mystery or confusion of material selection.

2 PLASTIC MATERIALS SELECTION

2.1 Polymers

In the selection of plastic materials for a commercial part design the first step is as in all technology development; that is, you must learn the basic definitions, concepts, and principles of that technology. Following will be a series of terms this writer thinks are significant for assisting one in the selection of materials for plastics part design: thermoplastics, thermosets, elastomers, polymerization reactions, characterization techniques, molecular weights and distribution, molecular structure of polymers, five viscoelastic regions of polymers, Carothers equation, and additives.

The starting point is with the definition of a polymer. A polymer is a compound consisting of repeating structural units. A simple example of a repeat unit is the—CH_2—chemical moiety. Two repeated units are equivalent to the organic compound ethane. Ethane is a gas at room temperature with a total molecular weight of 30 atomic mass units (amu). A polymer family with hundreds of thousands of these repeat—CH_2—units represents the polyethylenes with molecular weights in the millions.

A thermoplastic polymer is a polymer that consists of linear polymer chains. Whenever you use a thermoplastic, it is usually in its final molecular weight form. The major thermal event that one does is to process it into the final part form. There are three types of thermoplastics: amorphous, semicrystalline, and liquid-crystal polymers.

A thermosetting resin is one that contains a highly cross-linked polymer network when processed. One has to ‘‘cook’’ or cure the resin before it can be formed into its final shape.

An elastomer is a very lightly cross-linked polymer with the ability to be extend to a high elongation and snap back to its original dimensions when the forces has been removed.

Polymerization reactions play a usual role in the process of material selection. From the name of the polymer and its polymerization reaction, one can make a reasonable first attempt to select a plastic material. But the reader must be cautious with statements as the last one. These are general rules. Let us look at polyethylene as one example. Polyethylene is named from the monomer from which it is made. These monomers are polymerized through an addition reaction. Being formed through an addition reaction, the resultant polymers are water hating, or hydrophobic. For the first approximation this would be a good material to be put into a waterlike environment.

Now let us look at another polymer, polyethylene terephthalate. It is formed from the reaction of ethylene glycol with terephthalic acid or terephthalic acid ester. During the reaction in order for the polymer, polyethylene terephthalate, to build up molecular weights, it loses either water or alcohol as a by-product. The polymers formed from the type of reaction of adding two or more co-reactants under conditions of time, temperature, and other reaction conditions with the formation of a by-product such as water and alcohol are said to be formed by condensation and are named by the new chemical functional group formed. As a general rule of thumb these polymers are water loving, or hydrophilic.

Polyesters as mentioned are formed from the reaction of organic acids with organic alcohols with water as a by-product. Nylons are formed from the condensation reaction of organic acids with organic amines with water as a by-reaction. Polyimides are formed from the condensation reaction between acid anhydrides and organic amines with the release of water.

Knowledge of organic chemistry plays a very important role in the selection of a polymer for a plastic part. This writer is only posing general rules to the reader, and to the first approximation one can made very reasonable selection in the early stages of plastic part design using these general observations.

For example, if you were assigned to design a plastic part that had to exist in a water environment, your first choice could be an addition polymer such as polyethylene rather than a condensation polymer such as polyethylene terephthalate. The polyethylene is water hating, or hydrophobic. Thus it should not be affected by water.

This writer can already imagine the "but what about this incident" remarks. Yes, water bottles are made from polyesters. The bottles are dated for lifetime and the companies that fill these bottles with their clear spring mountain fresh water want you to see how pretty their water is. However, over time the polyester bottles will absorbed water. Water (sport) bottles that are used over and over and filled by the consumer are made from the addition polymers. They are also opaque. Transparency in these sport water plastic containers is not important. This polymer character of being transparent or opaque will be discussed later.

Molecular weight and molecular weight distributions are other important parameters for a polymer. The polymerization reaction is complicated. The polymerization reaction does not make a simple molecule. The reaction yields many different sizes of polymer chains. The molecular mass of each chain refers to its molecular weight. And as mentioned, since many different sizes of polymer chains are formed, there will be a distribution of the molecular weights.

Knowing the molecular weight and its distribution aids in the selection of polymers for a plastics part and in the lifetime of plastic part. The general techniques that can be used to determine molecular weights of polymers are achieved through viscosity measurements either in solution or using solid samples. Solution viscosities consist of timing the flow of polymer solutions of known concentrations through a fixed volume. Melt index, or melt flow index, is a standardized test in which the solid is used instead of a solution. A given amount of the polymer is heated to a certain temperature and a known force is applied to the molten polymer and its flow is timed. If all things are equal, the lower molecular polymer will flow through the given volume, the fastest thus revealing a low number. For the members of a given polymer family this is a reasonable way to distinguish between low- and high-molecular weight versions. The final technique is gel permeation (size exclusion) chromatography. The polymer is dissolved in a solvent. The solution is then passed through a series of tubes (columns) packed with different porous particles. As the solution passes through, the polymer chains with the highest molecular weights pass through the fastest. A detector measures the polymer chains as they exit the instrument. Thus one ends up with a chromatograph, which shows the distribution of the different molecular weights of the polymer chain in the sample. Solution viscosities are usually used by the polymer manufacturer. Melt flow index is used as an initial tool for material selection and as a tool to help determine the molding process. Gel permeation (size exclusion) chromatography was in the past treated as a research tool, but lately it has gained a great deal of popularity as a quality control technique.

Another important parameter for the different polymers refers to their thermal behavior. A typical thermoplastic is a solid at ambient temperatures. As the material is heated it starts to soften, then it flows and in some cases it melts. Then when it is cools down it solidifies. And depending of the container (mold) in which this is done, the thermoplastic will retain the shape of that mold. This process should be repeatable. Thus thermoplastics are recyclable. Another thermal property of the thermoplastics is creep. This property refers to the ability of the material to flow under a load as a function of temperature.

Thermosetting resin systems are quite different. When one processes a thermoplastic into a particular plastic part, its molecular weight has already been established at the manufacturer. With thermosetting resin systems, one starts with low-molecular-weight reactants and to process these ingredients you cure or ‘‘cook’’ the reactants into the desired final shape. If the reactants have been fully reacted, one ends up with one giant molecule. To process a thermosetting resin system into a part, the thermal events consist of heating the ingredients so they start to soften; then some of the ingredients melt. As the temperature is raised, the system is totally liquid. As the temperature continues to rise, the onset of curing (cross-linking) occurs. As the reaction proceeds, the viscosity increases and the part hardens. At the end of the curing reaction the part is solid; then it is cooled to ambient temperatures. Once formed the part cannot be reheated to change its shape. If a thermosetting resin system has been properly cured, it should not be affected by temperature or solvents.

The thermal behavior of elastomers is somewhat different that the thermoplastics and thermosets. As a first approximation it behaves more like a thermoplastic. We all know that car tires soften in the hot months of summer. And most elastomers will swell when placed into a solvent.

Thermal analyses are a set of techniques used to characterize the thermal behavior of the different type of polymers. In addition to informing one as to thermal characteristics of the polymers, they can assist in determining a processing cycle. Differential scanning calorimetry (DSC) yields the thermal events of a sample, i.e., melting points, onsets, maximums, and offsets of curing, decomposition temperatures, crystallization temperatures, and glass transition temperatures (this term will be discussed later). Thermogravimetric analyses (TGA) give the changes in mass of a sample as a function of temperature and environment. Thermal mechanical analyses (TMA) reveal the changes in volume of a sample (warpage and shrinkage) and glass transition temperatures. Dynamic mechanical analyses (DMA) provide the modulus and changes in modulus and glass transition temperature as a function of temperature, time, and oscillation (dynamic load).

The internal structure of the polymer will determine if it is transparent or opaque. This internal structure is referred to as polymer morphology. Thermoplastic polymers can be subdivided into amorphous, semicrystalline, crystalline, and liquid-crystal polymers. This classification is only reserved for thermoplastics. Morphology refers to how the polymer chains are arranged, in an ordered or disordered manner. Amorphous refers to total disorder. Crystalline refers to total ordered. Semicrystalline is a combination of disorder with domains of order within its structure. The liquid-crystal polymers refer to a special class of thermoplastics that retains its order in the melt. Based upon chemical principles as a material goes from the solid state to the liquid state, it goes from a state of order to one of disorder. The liquid-crystal polymers lack this transition, and this unique characteristic has an enhanced effect of the processing of these materials.

Let us now examine the internal structure of amorphous, semicrystalline, and crystalline thermoplastics. We have the two extremes—totally disordered in the arrangement of the polymer chains (amorphous) and at the other end total order (crystalline). Another way of looking at the arrangements involved is to view the polymer chains as spaghetti. We have cooked spaghetti (disordered) at one extreme and uncooked (ordered) spaghetti at the other extreme. Except for the liquid-crystalline polymers, most thermoplastic polymers are either amorphous or semicrystalline (a combination of polymer chains in ordered crystalline domains). Due to the presence of the crystalline domains, the semicrystalline polymers have a melting point, and light will be scattered as it hits these domains, thus giving the material an opaque appearance. Thus amorphous polymers do not have a melting point and are transparent.

The next important polymer definition or concept involves the five viscoelastic regions of polymers. If we plot the modulus of a thermoplastic material as a function of temperature, we obtain a graph such as the one shown in Fig. 1.

Region 1 represents the behavior of the material at low temperatures. It is in its glassy state. The mobility of the polymer chains has slowed down. The material is hard. As the material is heated, it comes through a transition to region 3. This region is known as the rubbery region and has lost in the range of three orders of magnitude of strength. As the sample is heated to an even higher temperature, the polymer (region 4) starts to decompose and finally at region 5 decomposition occurs with lose of strength.

A semicrystalline thermoplastic has the appearance of the dashed line in Fig. 1. The drop in modulus from the glassy region to the rubbery region is not as drastic with the semicrystalline polymers (region 2) as it is with the amorphous polymers. As the semicrystalline thermoplastic reaches its melting point, its strength falls off greatly and, as you would expect, goes from a solid to a liquid.

The transition between the glassy region of a polymer to its rubbery region is known as its glass transition and the temperature that it occurs at is its glass transition temperature.

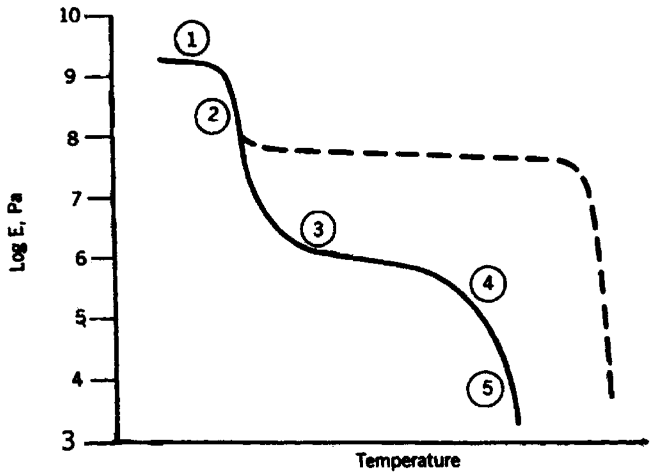

Fig. 1 Five viscoelastic regions of a linear amorphous polymer. The dashed line represents the behavior of a semicrystalline polymer (Ref. 2).

The glass transition is defined as "the reversible change in an amorphous material or in amorphous regions of a partially crystalline material, from (or to) a viscous or rubbery condition to (or from) a hard and relatively brittle one."[1]

Some individuals use the term glass transition temperature while discussing cured thermosetting resin systems. To this writer, if the thermosetting resin is completely cured, it should not have a glass transition temperature. If it is completely cured as you heat the material over a temperature range, it should be unaffected by temperature until it reaches its decomposition temperature. If you perform an analysis and observe a glass transition temperature, it is either because the thermoset is not being completely cured or because of the thermoplastic nature of the cross-linked network. If you perform a thermal technique to determine glass transition temperature, cool the sample to ambient temperature and repeat the analysis on the same sample. If the material is not fully cured, the repeat run should indicate a higher apparent glass transition temperature and a lower drop in modulus.

Figure 2 shows a comparison between the glass transition temperature and the melting point of a thermoplastic polymer. The key feature of the graph shows

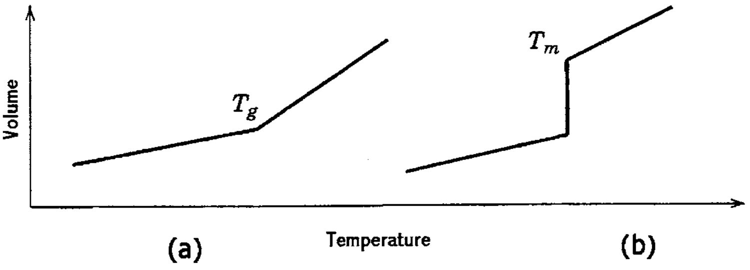

Fig. 2 Comparison of the glass transition temperature (*a*) to a melting point (*b*) of a thermoplastic polymer (Ref. 2).

that as the material reaches its melting point that is a discontinuous in its volume. In the case of the glass transition temperature its rate of volume changes with temperature. As one passes through the polymer's glass transition, its thermodynamic properties change. Thus, in selection of a thermoplastic for a plastic part, it is always best to use the material above its glass transition temperature. There are exceptions to this rule. For example, polymers like the polyethylenes are used in their rubbery region due to their subambient temperature glass transition temperatures.

This writer is including the Carothers equation in the discussion of the selection of polymeric materials for plastic molded parts. The equation is relatively simple, but the impact of it on a thermoplastic polymerization is very critical:

$$\overline{X}_n = \frac{2}{2 - pr}$$

The term $\overline{X}_n$ refers to number-average degree of polymerization, p is the extent of reaction, and r either indicates the ratio of reactants or purity of the reactants. Basically, what this equation tells us is that one needs a high conversion of pure reactants and the correct stiochiometry to obtain the proper molecular weight. Small changes in purity, incomplete reaction, and incorrect ratio of reactants can have a drastic effect on the moldability of your part or its performance behavior. Thus consistency and repeatability of the molded part you shipped to your customer is highly dependent upon the consistency and repeatability of the materials from your polymer manufacturer, your compounder, and your molder.

2.2 Plastics

Plastics are simply polymers with additives. These additives perform many different functions. Some refer to these materials as "foo-foo" dust. The addition of additives to the polymer enhances the process of the making of the part, the product performance and lifetime of the part, and the appearance of the part. A partial list of these additives include antioxidants, light stabilizers, acid scavengers, lubricants, polymer processing aids, antiblocking additives, slip additives, antifogging additives, antistatic additives, antimicrobials, flame retardants, chemical blowing agents, cross-linking and controlled degradation of polyolefins, colorants, fluorescent whitening agents, fillers, nucleating agents, and plasticizers.

From the name of the additive one can figure out the function of the additive. There are exceptions to this, and in most cases we do not know what the additive package compounders put into a polymer. This information is treated as confidential. Thus one must be careful when one switches material from one supplier to another. Even though the starting polymer may be same, different additive packages will affect the performance behavior and lifetime of the designed plastic part. Thus once a material has been selected, this writer highly recommends that one completely characterize that material in case your supplier changes the material or its consistency changes or you are involved in determining product failures. As a consultant, this writer has been involved in several failure analyses projects and the worst thing that happens is that there is no baseline material data. These problems are difficult and expensive to solve without baseline data.

Two types of additives will be defined in this section due to their importance. These are fillers and plasticizers. Fillers are added to polymers to affect the color and smoothness properties of the final molded part, to assist in the molding of the part by changing the flow behavior of the plastics, and lastly to reduce the cost of the molded part.

Plasticizers are unique materials. They are added to a polymer to reduce the hardness of the polymer or make it more flexible. A good example of a plasticizer is the "smell" we experience when we purchase a new car. The new car smells comes from the plasticizer and the seats are nice and soft. Over time the smell is gone, the seat becomes hard and brittle, and we have to clean the inside of the windshield. The reverse of this can occur with hydrophilic polymers in the presence of water. The polymer absorbs water, its glass transition temperature drops, and the material becomes softer. In the case of hydrophobic thermoplastic materials oils will have a similar effect.

Thus, it should be noted that these plastic systems are dynamic and they are in a constant change. If you design a plastic part for a lifetime of 5 years, it would be "nice" to test the part for 5 years under the operating conditions of the plastic part assembly. However, based upon "time to market" that is not an option. One can retain samples of production.

2.3 Reinforced Plastics

Reinforced plastics are plastics containing reinforcing elements within a plastic matrix. There are many different types and forms of reinforced plastics. And whenever one refers to reinforced plastics, they are referring to thermoplastics reinforced with either long or short discontinued (chopped) fibers, and the parts are manufactured by injection molding. Reinforced plastics are in essence forms of composite materials. A composite is a heterogeneous mixture of matrix resin, reinforcement, and other components that act in concert with each other. The matrix resin protects the reinforcement from itself due to wear and it "glues" the reinforcement in place. The reinforcement provides strength to the plastic part and enhances the properties of the matrix resin. Also the reinforcement helps dissipate the energy throughout the structure when impacted.

Properties of reinforced plastics should be obtained from the supplier. There are many variations of reinforcement forms and sizes.

3 PLASTIC MATERIALS SELECTION TECHNIQUES

It is difficult to discuss how one starts to select plastic materials for a particular design. At first the tenacity is to hope that you doing a second version of a previous designed plastic part. This is wishful thinking. Second one hope that one is exposed to an individual in their organization whom has experience in plastic material selection. Or, if you are really fortunate, you have a materials engineer in your organization that knows the material science of plastic materials.

This writer's first experience in selecting materials was relatively simple. The criteria were basically set by the equipment available for the project. Then criteria such as the temperature, chemical requirements, and the number of parts needed are used to pick the material. An eight-step criteria was then developed. The criteria were functionality, chemical resistance, external processing (your supplier), internal processing, lifetime, design margins, cost, and greenness of the part. As the demands on the materials increased, subcriteria to the criteria were developed and added to the list and is represented by Table 1.

The above scheme works reasonably well. But it does not include the most important factor in a material selection. That factor can be represented with such words or phrases like *timing, schedule,* and *time to market.*

4 PLASTIC JOINING TECHNIQUES

Table 2 contains a list of most of the different techniques used to join two plastic parts together. Each technique has its own unique advantages and limitations. This writer has more experience with adhesive bonding. In the selection of adhesives one can follow most of the criteria and subcriteria as listed in Table 1.

In the adhesive bonding of plastic parts together there are several other issues that you should take into account. One refers to the failure mechanism that you want to strive for in your design. There are three failure mechanisms for an adhesive joint. They are adhesive, cohesive, and substrate failures. An adhesive failure is failure of adhesive joint at the interface. Cohesive failure is failure within the adhesive and finally substrate failure of the plastics. This is failure within the plastic parts being joined together. Failure within the plastic parts would be the best case. And with surface preparation techniques, especially with treatments involving silane coupling agents, this failure mode is not unrealistic as observed by this writer.

The golden rule of design using adhesives was (and is) to design to at least cohesive failure.

One chemical principle that you should take into account in the selection of adhesives is ‘‘like dissolves like.’’ It translates into the more similar the adhesive is to the plastic you are trying to bond together the stronger the bond.

5 PLASTIC PART DESIGN

In this section, I have to be honest and admit that I do not know how to design plastic parts. I cannot draw a straight line with a ruler. I rely on the suppliers’ design guides and the two design books (Malloy and Dym) listed in the Suggested Readings. And I am also fortunate to have a good network that is only a phone call away. However, I do perform failure analyses on molded plastic parts, and there are tricks I employed during an analysis. One technique is to perform an ashing of a plastic molded part. One can obtain information as to the flow of the plastic and fiber orientation if the part is reinforced.

One can also section the molded reinforced plastics into smaller specimens that can be analyzed by TGA to determine percent resin and reinforcement contents; this in turn will show you the consistency of the molded part.

Sometimes if the plastic has a long distance to flow in a mold, the polymer chains can separate. The smaller polymer chains travel faster than the larger chains. To verify the consistency of the polymer molecular weight throughout the molded plastic part, one can section the part and subject the specimen to gel permeation chromatography.

6 PLASTIC PART MATERIAL SELECTION STRATEGY

After reading the first part of this chapter one may be either totally confused or have a headache. This writer understands perfectly. Materials selection is not an

Table 1 Eight-Step Criteria for the Selection of Materials (Thermoplastics, Thermosets, Elastomers, and Adhesives)

Main Criteria	Subcriteria
Functionality	Purpose of part Type and magnitude of normal service stresses Loading pattern and time under load Fatigue resistance Overloads and abuse Impact resistance Normal range of operating temperatures Maximum and minimum service temperatures Electrical resistivity Dielectric loss Antistatic properties Tracking resistance Flammability Surface finish Color matching and color retention Tolerances and dimensional stability Weight factors Space limitations Allowable deflections
Materials acceptance	Compatibility with chemicals Solvent and vapor attack Reactions with acids, bases, water, etc. Water absorption effects Ultraviolet light exposure and weathering Oxidation Chemical erosion and/or corrosion (electrochemical effects) Attack by fungi, bacteria, or insects Leaching of additives from the part material into its environment Absorption of components into the part from its environment Permeability of vapors and gases Normal range of operating temperatures Maximum and minimum service temperatures
Environmental concerns	Scrap rates Recyclability Chloro- and fluoro- polymers
Lifetime	Product lifetime Reliability Product specifications Acceptance codes and specifications
Margin	Safety design factors
Internal process	Normal range of processing temperatures Maximum and minimum processing temperatures Choice of processes Method of assembly Secondary processes Finishing and decorating Quality control and inspection Contamination
External process (supplier of parts)	Normal range of processing temperatures Maximum and minimum processing temperatures Choice of processes Method of assembly Secondary processes Finishing and decorating Quality control and inspection Contamination Timing for part design changes Timing for prototype molds Timing for production molds

Table 1 (*Continued*)

Main Criteria	Subcriteria
External process (supplier of parts) (*Continued*)	Technical support from supplier Contamination
Cost	Materials costs Materials availability Alternative material choices Suppliers availability Part costs Cost of capital plant: molds and processing machines Operation costs of component including manufacturing and fuel consumption Capacity

easy task. Following represents a series of suggestions and hints that hopefully make the task easier.

First, if you are fortunate to be part of a large organization, develop a team of individuals that you think may help you. This writer has been in favor of at least a four-person team. The team should consist of a design engineer, a materials type (analytical chemistry people in one's organization may be a good substitute if you have no true materials engineer), an internal experience engineer, and a representative from the procurement department or an internal company buyer.

Some of the selections of the team are obvious. Each can handle part of the criteria and subcriteria as listed in Table 1 or any other type of list of requirements that you develop.

The first step is probably the most difficult. That is the selection of the polymer families to evaluate. Initially, some consult the *Modern Plastic Encyclopedia*. I am partial to Domininghaus's book on *Plastics for Engineers* for the selection of a thermoplastic. The *Modern Plastic Encyclopedia* is an excellent and well-respected source book. However, it only contains one data point within the total history of a particular thermoplastic. Domininghaus's provides pressure–volume–temperature (P–V–T) graphs on the various thermoplastic families. That type of data is significant in the processing of materials. These publications are listed in the Suggested Readings.

Table 2 Plastic Joining Techniques

- Adhesive bonding
- Electrofusion bonding
- Friction welding
  - Linear
  - Rotational
- Heated tool welding
  - Hot plate
  - Hot shoe
- High-frequency welding
- Hot gas welding
- Induction welding
- Infrared welding
- Laser welding
- Mechanical fastening
  - Beading
  - Hot stakes
  - Interface fits
  - Molded-in and ultrasonic inserts
  - Molded-in threads
    - Riveting
    - Self-threading screws
    - Snap fit
- Solvent joining
- Thermal impulse welding
- Ultrasonic welding

Now the next action is to obtain samples and technical information from the suppliers. As previously mentioned, most suppliers have design guides for their polymers. These are an excellent source of information that can be helpful in your efforts to design the plastic part. Also, obtain from the polymer supplier any analytical procedures as to how they characterize their materials. This will assist your internal analytical people to develop a material knowledge database.

Next, you and your team should review all the available data on the polymer under consideration. A good literature search through a technical library may save you time, effort, and money. In addition, you may want to perform your own tests to fill in missing information. The polymer supplier can be helpful in this area. The supplier can provide molded, American Society for Testing and Materials (ASTM) test coupons that you can use for your own testing. If you are designing a plastic part to be in a certain chemical environment, you may wish to test your selection in a chemical soak-type test. This can be achieved by soaking a test coupon in the chemical of concern. You may also want to soak the test coupon at different temperatures within the operating range of the designed plastic part or within a linear range of behavior of the polymer. If you can perform such a chemical soak at three different temperatures, you can predict the lifetime of the polymer if it can be related to a chemical failure. However, data of this sort must be obtained using the principles of chemical kinetics.

An example of aging a part could be found within the different outcomes (failure mechanisms) of an egg. Take an egg and set it on a shelf and leave it alone. After several months the egg becomes rotten. Take a similar egg and place it under a hen and after a while we had a cute little chick. As a last example, take another egg and place it in boiling water and after about 10 minutes you have a hard boiled egg. Never pick temperatures throughout the viscoelastic region of a thermoplastic; you will obtain three different responses (at its glassy region, at its glass transition temperature, and at its rubbery region) of the material.[3]

Now we can proceed with the plastic(s) of choice. This is the polymer with the magical foo-foo dust that the compounder puts in it for various reasons. Chemical soak tests are extremely important in the cases where the plastic part is used in a chemical environment. We do not want anything from the plastic to be extracted into the ‘‘chemical environment’’ thus either affecting the properties of the plastic or contaminating the chemical environment. And the reverse is also true; we do not want the plastic to absorb chemicals from the chemical environment. This could cause the properties of the plastics to be lower due to a plasticization effect.

Also the same type of chemical soak tests should be conducted on the final molded part with the chemical soaking to an exposed portion of the design. In addition to develop a material knowledge base for the particular part you are designing, you will need data in the event you or others have to perform failure analysis on the molded part.

You may request some of the data mentioned in this chapter from your suppliers.

At this stage you should hopefully be dealing with the molded plastic part assembly. The next part is to design functional tests that reflect the functionality of the plastic molded part assembly as it leaves your facility and as it performs in the field.

7 CONCLUSION

This chapter was written as hints, suggestions, and "tricks" to assist one in the selection of materials for plastic parts design. These hints, suggestions, and tricks have helped this writer in various industrial positions held and in consulting projects completed.

Like everyone else I have experienced the phase "we don't have time to do it right the first time, but we have time to redo it." It is always easier to do some right the first thine.

I have participated in projects in which millions were spent to develop a plastic assembly and the project failed for the stubbornness of not spending a couple of thousands of dollars on an analytical test.

I have heard words like "mechanical engineers can pick materials." This may be true. But I have again experienced projects where looking in a polymer handbook would have saved thousands of dollars and many months of work. Case in point, polyethylene terephthalate was selected as the material of choice for a plastic part that had to withstand an internal processing step of being adhesively bonded to another part for 2 min at 150°C. The grade of polyethylene terephthalate used was a recycled grade with a glass transition temperature in the vicinity of 60°C. Placing the final assembly in an oven at 60°C to simulate an aging test failed all parts due to changes in dimensions of the part. Subjecting a polyethylene terephthalate coupon to 60°C testing or checking the literature would have been helpful.

Another example was the use of polystyrene as a throw-away coffee cups. Several years ago a fast-food chain was sued for injuries a customer suffered for drinking coffee from one of these cups. Part of the injuries occurred because the cup was made from polystyrene. Polystyrene has a glass transition temperature in the vicinity of 100°C. This temperature is the same as the boiling point of water. If the coffee is extremely hot, it could reach temperatures close to its glass transition temperature or in the vicinity when the material starts to transit from the glassy region to the rubbery region of the polymer. In one particular case it did, thus losing its structural integrity, causing the coffee to spill out of the cup, burning the customer, and resulting in a legal action.

In the conclusion I would like to say that in writing this chapter I have been fortunate to have the benefit of books, good teachers, a good network of suppliers, and good co-workers who were part of my team. Engineers who were willing to learn, and management that had faith in my methods, and, most important, my willingness to be taught by others. I wish you the same.

SUGGESTED READING LIST

As mentioned earlier, this chapter was written from the short courses and graduate lectures that this writer has given in the past. However, I feel obligated to provide the reader with a reading list. The amount and nature of the information is even overwhelming to the author.

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