

**Safety Guide 100**

**DESIGN GUIDE FOR PACKAGING AND OFFSITE TRANSPORTATION  
OF NUCLEAR COMPONENTS, SPECIAL ASSEMBLIES, AND RADIOACTIVE  
MATERIALS ASSOCIATED WITH THE NUCLEAR EXPLOSIVES  
AND WEAPONS SAFETY PROGRAM**

**CHAPTER 10.0**

**MATERIALS AND FABRICATION**

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## ACRONYMS

AISI	American Iron and Steel Institute
ASTM	American Society for Testing and Materials
APA	American Plywood Association
AMS	Aerospace Materials Specifications
ALCOA	Aluminum Company of America
CS	Commercial Standard
DBT	Ductile-to-Brittle Transition Temperature
DOD	Department of Defense
EPM	Ethylene Propylene Monomer
EPDM	Ethylene Propylene Di-Monomer
HAC	Hypothetical Accident Condition
HSLA	High-Strength Low-Alloy
HDPE	High Density Polyethylene
HMW	High Molecular Weight
HTLA	Heat-Treatable Low-Alloy
LDPE	Low Density Polyethylene
MDPE	Medium Density Polyethylene
MWD	Molecular Weight Distribution
NCT	Normal Conditions of Transport
PFAT	Perfluoroalkylenetriazine
PIR	Polyisocyanurate
PTCF	Polyisocyanurate
RTV	Room Temperature Vulcanized
SAE	Society of Automotive Engineers

SNM	Special Nuclear Materials
TPO	Thermoplastic Olefins
UHMWPE	Ultrahigh Molecular Weight Polyethylene
VDF	Vinylidene Fluoride
VLDPE	Very Low-Density Polyethylene

## **10.0 MATERIALS AND FABRICATION**

### **10.1 INTRODUCTION**

#### **10.1.1 Scope**

This chapter specifies preferred materials and fabrication methods in the design of packages to transport special assemblies, components and radioactive material associated with the nuclear explosives and weapons safety program.

#### **10.1.2 Approach**

The functions of each package design, the preferred materials that will accomplish those functions, and information for each of the preferred materials for each function are given. Included in this information are experiences of packaging organizations using the preferred material or fabrication method. Materials that are not on the preferred materials list may be used to make the packaging if justification for their use is properly documented.

#### **10.1.3 Design Process**

The information in this chapter is intended to aid the packaging designer in selecting materials and fabrication methods for the packaging.

Materials will be selected for their ability to satisfy the structural, thermal, subcriticality, shielding, and compatibility requirements. The availability and cost of materials and the fabrication costs will also be considered.

The selection of fabrication methods will be based on the methods that will produce the desired component (using the selected materials) at a reasonable cost. The availability of fabrication equipment and the properties of the fabricated components must also be considered.

#### **10.1.4 Definitions (Chapter Specific)**

- *Confinement* - the holding together of the packaging during all normal conditions of transport and hypothetical accident conditions so the packaging components can perform all other functions.
- *Containment* - the retention of the radioactive material during all normal conditions of transport and hypothetical accident conditions.
- *Impact Limiter* - packaging components that will absorb part of the energy of a collision so that containment or other vital components are maintained throughout the hypothetical accident.
- *Shielding* - the blocking of the nuclear radiation from the content such that the radiation level at the surface of the packaging and at 1 meter from the surface of the packaging is acceptable during all normal conditions of transport and hypothetical accident conditions.
- *Special Nuclear Material*—DOE 5632.1A, Attachment 2, p. 13).

1. Plutonium, uranium enriched in the isotope 233 or in the isotope 235, and any other material that, pursuant to the provisions of Section 51 of the Atomic Energy Act of 1954, as amended, has been determined to be special nuclear material, but does not include source material; or
  2. Any material artificially enriched by any of the foregoing but that does not include source material.
- *Subcriticality* - the function of packaging components that will ensure that the content remains subcritical during all normal conditions of transport and hypothetical accident conditions.
  - *Thermal* - the function of the packaging components that will ensure that thermal conditions will result in the survival of a package during normal conditions of transport and hypothetical accident conditions and will properly dissipate radioactive decay heat.

## **10.2 MATERIALS**

### **10.2.1 Content Materials**

The scope of this document is limited to packagings designed to transport some of the following SNM in Table 10.1.

In addition, the content may include small amounts of other radioactive materials, such as fission products and nonradioactive material, limited in amount only by the capability of the packaging to meet regulatory requirements.

**Table 10.1. Content materials**

<b>Material</b>	<b>Form</b>	<b>Sources of Nuclear and Physical Properties</b>
$^{233}\text{U}$ , $^{235}\text{U}$ , $^{238}\text{U}$	Solid (parts, powder, chips, etc.); pure metal, oxides, nitrides, other compounds	C. V. Parks, ed., <i>SCALE: Material Information Processor for SCALE</i> , Volume 3, Section M7, Rev. 4, NUREG/CR-0200, September 1981.
$^{239}\text{Pu}$ , $^{240}\text{Pu}$	solid (parts, powder, chips, etc.); pure metal, oxides, nitrides, other compounds	C. V. Parks, ed., <i>SCALE: Material Information Processor for SCALE</i> , Volume 3, Section M7, Rev. 4, NUREG/CR-0200, September 1981.
Tritium	solid, liquid, or gas (mixtures and compounds)	<i>Health Physics Manual of Good Practices for Tritium Facilities</i> , MLM-3719, Draft, EG&G Mound Applied Technologies, December 1991.

## **10.2.2 Packaging Materials**

### **10.2.2.1 Requirements for packaging materials**

The materials used in an SNM packaging must satisfy the following functions.

- Packing
- Containment
- Thermal
- Subcriticality
- Shielding
- Impact limiter
- Confinement

No single material will accomplish all of the functions; however, some materials will contribute to more than one functional need. A list of preferred materials has been selected based on the experience of various packaging organizations. The criteria for selection were capability, availability of the material, availability of design information, and cost. A sample packaging showing the function(s) of each of the components is shown in Fig. 10.1. A listing of preferred materials and the functions that they will satisfy are given in Table 10.2.

Materials other than the preferred materials may be used for SNM packagings. However, justification must be properly documented. The following paragraphs discuss the characteristics of the materials that will accomplish each of the needed functions.

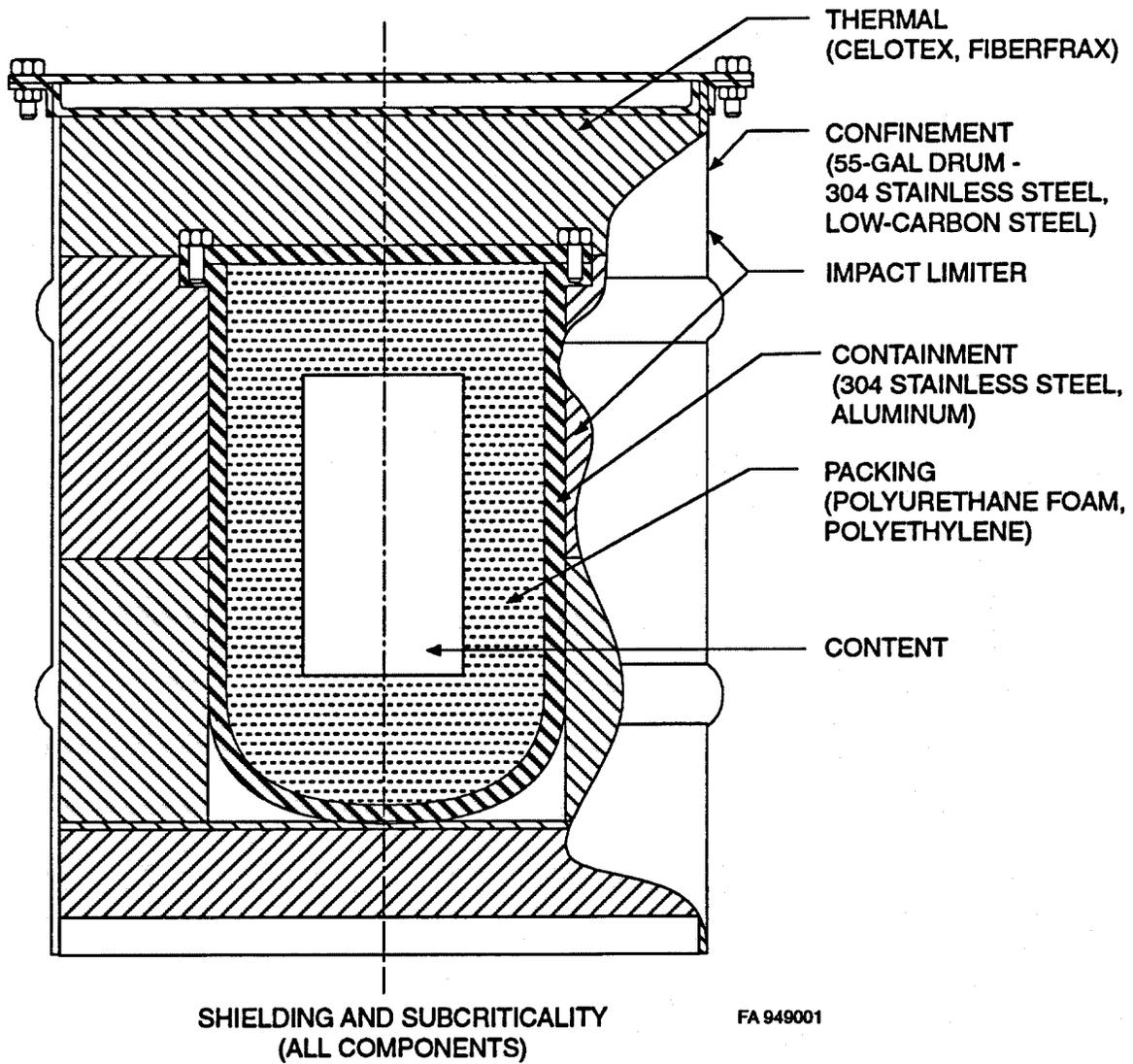


Figure 10.1. Sampling package.

**Table 10.2. Preferred Materials and Their Functions**

Preferred Material	FUNCTION									
	Containment	Thermal	Criticality	Shielding	Impact Limiter	Confinement	Packing			
Austenitic Stainless steel	X			X	X	X				
Carbon Steel	X			X	X	X				
High-Strength Steel	X					X				
Aluminum Alloy 6061	X					X	X			
Insulating Board		X								
Ceramic Fiber Materials		X								
Fir Plywood		X								
Redwood					X					
Urethane Foam		X			X		X			
Silicone Foam		X			X		X			
Fluoroelastomers	X									
Neoprene	X									
Ethylene Propylene	X									
Polyolefins							X			
Silicone Elastomers						X				

**Containment.** The containment boundary(s) must prevent the release of radioactive materials during both normal conditions of transport (NCT) and hypothetical accident conditions (HACs) at a rate higher than that allowed by regulation. The walls, heads, welds, fasteners, and seals that make up the containment boundary(s) must be capable of withstanding the mechanical, thermal, and physical conditions that may be placed upon them.

**Thermal.** The characteristics of the materials used must accommodate the thermal loads that may be applied to the packaging. Thermal loads can include radioactive decay heat from the contents, heat from the HAC fire, and heat from the sun. The environment can also affect the thermal load on the packaging. Various combinations of thermal loading can occur during the shipment of a package. The thermal properties of the materials used and the configuration of those materials must be such that thermal, containment, subcriticality, and shielding are maintained.

**Subcriticality.** Subcriticality must be maintained during all NCT and HAC. In many cases, subcriticality is dependent on keeping all moderators (most likely water) out of the inner container and maintaining adequate spacing between multiple containers. Consequently, if containment and confinement are maintained, subcriticality will be maintained (see the characteristics of the materials used to maintain containment and confinement).

**Shielding.** The radiation dose rate at the surface and one meter from the surface of the packaging must not exceed regulatory limits during both NCT and HAC. If the packaging is deformed during HAC, consideration must be given to the possibility that the radiation source may be nearer to the surface of the packaging and, consequently, the radiation dose rate at the surface will be higher after deformation. In some cases, the packaging design that satisfies the functions other than shielding may provide some shielding. In other cases, the materials used or the thicknesses of the materials used will have to be

changed to provide adequate shielding. In addition to transportation, the packaging may be required to meet more stringent facility dose rates. These lower criteria must be considered during the material and fabrication selection.

**Impact Limiter.** The components of the packaging that act as an impact limiter are usually those components at or near the outer surface of the packaging. In the sample packaging (Fig. 10.1), the stainless steel drum and the thermal material act as impact limiters (they also perform thermal and confinement functions). The impact limiters absorb energy from the HAC drops so that the other functions of the packaging are preserved.

**Confinement.** The components of the packaging that provide confinement must hold the package together during HAC to the extent that the other functions of the packaging are fulfilled. Usually, the confinement components are those that make up the outer surface of the packaging. In the sample packaging (Fig. 10.1), the stainless steel drum is the confinement component. During the drop-test portions of HAC, the extent of the deformation and opening of the drum must be such that allowable radiation doses are not exceeded and containment is maintained during the fire test.

#### **10.2.2.2 Properties of preferred materials**

This section discusses how the properties of each of the preferred materials give the material the ability to accomplish the needed functions given in Subsect. 10.2.2.1.

#### 10.2.2.2.1 Austenitic stainless steel

The most common austenitic stainless steels are perhaps from the Type 304/304L and Type 316/316L families of alloys. Together, these alloys are the work horses of the chemical process industry because of excellent general corrosion resistance, ease of fabrication (including forming and welding), good combination of strength and toughness, oxidation resistance, availability of standards and specifications, and alloy availability in a wide variety of shapes and sizes. Type 304L is an austenitic iron-base alloy nominally containing 18% chromium, 8% nickel, and a carefully controlled (0.03% maximum) carbon content. The "L" designation distinguishes the low-carbon grade from the nominal carbon content grade of 304 (that may contain up to 0.08% carbon). Types 316 and 316L stainless steel are similar to 304 and 304L in composition except that the former includes nominally 2% molybdenum. The addition of molybdenum enhances localized corrosion resistance in many aqueous media but, depending on product form, may add considerable expense compared with 304/304L alloys. The cast equivalents for these materials are CF-3 (304L), CF-8 (304), CF-3M (316L), and CF-8M (316). [The cast materials do not have exactly the same composition as their wrought counterparts and typically have a duplex structure (ferrite in an austenite matrix) as opposed to a fully austenitic structure.] None of these alloys can be hardened by heat treatment, but each work hardens readily.

Based on composition specifications and the allowable ranges for all alloying elements, the only difference between Types 304 and 304L and between Types 316 and 316L is the carbon content. The small difference in carbon content has a rather insignificant affect on most physical and mechanical properties, but it can have a profound influence on corrosion resistance in some environments. The reason is that welding thermal cycles or other heat treatments that include the 500° to 800°C temperature range, even for relatively short durations, tend to precipitate chromium carbides on the alloy grain boundaries. These carbides deplete the surrounding material of chromium and the alloy loses its

"stainless" character on a local basis. As the alloy carbon content is lowered, the rate and amount of carbide precipitate forming in the 500° to 800°C temperature range decreases. With less than 0.03% carbon, Types 304L and 316L have very good resistance to potential carbide precipitation problems.

Otherwise, the influence of carbon content on the properties and performance of these alloys is minor. For example, mechanical properties (tensile and yield strengths) of solution treated Types 304 and 304L are essentially identical (some sources indicate that Type 304 is a few percentage points stronger at low temperature, but the difference decreases rapidly as temperature is increased). Compared with Type 304, the lower carbon content of Type 304L improves significantly the notch toughness of welds (particularly at low/cryogenic temperature) and decreases work hardening somewhat. Creep resistance of Type 304 is somewhat higher than that of Type 304L under some loading conditions. Physical properties of Types 304 and 304L, such as thermal conductivity and coefficient of expansion, are considered essentially identical for these alloys.

The same trends between properties of Types 316 and 316L apply, and in general, there are no significant property differences between the Type 304 and the Type 316 alloy families. One notable exception is that compared with Types 304/304L, Types 316 and 316L are particularly prone to embrittlement during heat treatment or other thermal cycles that involve the temperature range of 650° to 850° C. In addition to carbide precipitation (discussed earlier, temperature range for which overlaps this range), sigma phase formation can be very detrimental to ductility and toughness. Sigma phase forms most readily from the ferrite phase (which is present in most welds of austenitic stainless steels) but forms very slowly at austenite grain boundaries, too. When molybdenum is present, as in Types 316/316L, the rate of sigma formation can be very rapid at either location. A practical problem for container materials in this regard is the 30-minute 800° C "thermal test" following drop tests. The possibility of embrittled welds of Types 316 or 316L is substantial after this heat treatment.

As a result of the similarities in physical and mechanical properties, with a clear advantage for "L" grades in terms of resistance to corrosion problems induced by welding or other thermal cycles, the use of type-standard carbon content grades is discouraged in favor of "L" grades for most critical containment materials. Furthermore, because of increased cost and the likelihood for embrittlement under some conditions for Types 316/316L compared with Type 304L, selection of materials other than Type 304L is further discouraged.

A particularly exhaustive reference including extensive text on fabrication and design practices and data tables for physical and mechanical properties of stainless steels, including Type 304L, can be found in Refs. 1-3.

#### **10.2.2.2.2 Carbon steel**

The primary strength of plain carbon steel as a material of construction is that it is inexpensive and can be processed in a wide variety of forms to develop a broad range of physical and mechanical properties. Its primary weakness is that it has very limited corrosion and oxidation resistance and can be prone to brittle behavior under some service or processing conditions. Plain carbon steels are iron-based alloys with specific carbon contents and only small amounts of other elements that are necessary in the steel-making process; no intentional alloying elements are introduced. Typically, the designation of plain carbon steel is a four-digit number of the form 10XX, where XX represents the nominal carbon content of hundredths of a weight percent (0.00XX).

Many attempts have been made to generate a standard way of classifying and designating steels. Basically, these classification schemes group the steels by chemical analysis or composition, mechanical properties, or use. Typically, the methods based on mechanical properties and use are related to specific

industries. A large number of methods are based on chemical analysis. Probably the most commonly used designations in the United States are those developed by either the American Iron and Steel Institute (AISI) or the Society of Automotive Engineers (SAE). These designations are essentially the same; both use a four-digit code for both plain carbon and low-alloy steels. Sometimes, the AISI code is preceded by a letter indicating a particular method of manufacture.

Another set of designations frequently used are those developed by the American Society for Testing and Materials (ASTM), which issues individual specifications, each of which covers a specific steel form (i.e., bar or plate) or use. In the case of steels, these specifications are all preceded by "ASTM A" and include a number indicating the specification, which may also include subsequent letters or numbers that further subdivide the materials by analysis. For this purpose, the ASTM code frequently includes the AISI designation. The complete ASTM code also includes the year of origin and may contain a "T," indicating that the specification is tentative. Plain carbon steels (such as AISI 1015) have a carbon content ranging from 0.05 to 0.2 wt % and could be termed "low-cost, low-strength, low-carbon steels." If the impurity levels (especially phosphorus, sulphur, and oxygen) are kept low, these steels can exhibit good weldability because of their corresponding low hardenability and can also have good ductility. Typical uses include ship and boiler plate; pipe; reinforcing bars; and corrugated or deep-drawn sheet.

Plain carbon steels such as AISI 1030 (0.3 wt % carbon) with nominal carbon contents ranging from 0.25 to 0.45 wt % are considered medium-strength, low-cost steels. They are hardenable by thermal treatment and cold working to a degree proportional to their carbon content. Properties are also controlled by the amount of impurities that tend to assist in hardenability while decreasing ductility. Silicon and manganese, which have little, if any, effect on properties, are added to some extent in all of these steels to help control oxygen and sulphur content during manufacture.

Carbon steels with medium high carbon-content ranging from of 0.5 to 0.8 wt % are typically used where high hardness is required but good ductility and toughness are not. These materials are likely to be suitable for container components. These materials are readily available in the form of sheet, bar, tube, castings, and forgings, but they are not usually found in thin sheet or tubing because the forms require frequent annealing to avoid embrittlement during manufacture, which increases the cost.

For a given plain carbon steel, its properties and suitability for certain uses are controlled by thermal treatment, working condition, and foundry practice. Typical heat treatments for plain carbon steels involve heating the material above the transformation temperature (the temperature at which all constituents are in a single-phase, solid solution called austenite) and cooling rapidly (such as quenching in oil or water to form a supersaturated solution that is typically strong, hard, and brittle) or cooling slowly in a closed furnace or air (normalizing) to produce a softer, weaker, more ductile material. Stress relief heat treatments and tempering heat treatments are performed after quenching to restore some ductility and toughness to a hardened steel at the expense of some strength loss. Scale removal by grit blasting or pickling may be required after thermal treatments.

The choice of fabrication temperatures or practices influences mechanical properties, machinability, and dimensional stability. In addition to hardening by heat treatment, steels can also be work hardened through plastic deformation at or near ambient temperatures. This procedure, called cold working or cold finishing, increases strength and hardness but decreases ductility and toughness. Cold working requires more power because the material work hardens as it is formed. Intermediate annealing to recrystallize and soften the material may be required with cold forming. However, cold forming is a dimensionally precise fabrication method that can produce a readily machined part without the problems associated with oxidation. Hot working is performed above the transformation temperature of the steel so that recrystallization occurs simultaneously with plastic deformation, preventing work hardening. Hot

forming requires less power but makes final dimensions less precise and can cause surface problems because of the oxidation/corrosion products formed.

Foundry practice, particularly the extent of deoxidation, can also influence mechanical properties of steel. Rimmed steels are only slightly deoxidized, allowing some evolution of gas (produced by available oxygen reacting with carbon) to occur during solidification. As a result, the outer skin of the ingot is depleted in carbon. If the rimming action is controlled and if the carbon and manganese contents are not too high, the surface that is produced can be ductile. Thus, plain carbon steels are specified as rimmed steels that are suitable for processes such as cold rolling. Killed steels are fully deoxidized (usually by additions of aluminum or silicon), and they are typically chemically homogeneous with uniform mechanical properties and improved low-temperature toughness. The uniformity of killed steels makes them most suitable for applications involving hot-forging, cold extrusion, carburizing, and thermal treatment. However, killed steels are not particularly suitable for applications where especially fine surface finish is important. Semikilled steels are not fully deoxidized, but they offer slightly better surface properties. They are suitable when neither the surface quality and cold forming attributes of rimmed steels nor the uniformity of killed steels is required. Capped steel cools with some concurrent oxygen evolution (not as much as with rimmed steel) and has poor homogeneity, but it has good surface properties. Capped steels are used primarily for cold forming.

Plain carbon steels, almost independent of heat treatment condition, experience a ductile-brittle transition as the temperature is lowered. Often, the transition is marked by a very significant decrease in notch toughness as the temperature decreases. Many factors influence the ductile-to-brittle transition temperature (DBT) and whether it is a sharply defined value, typically in the range of  $-20^{\circ}$  to  $+20^{\circ}\text{C}$  for a typical 1040 steel, and notch toughness may decrease from maximum to near zero over a  $40^{\circ}$  to  $50^{\circ}\text{C}$  drop in temperature.

A thorough summary of applicable steel specifications is given in the Ref. 4. This reference categorizes the plain carbon steels by carbon content and provides a good summary of some representative properties for each group, including the following:

- Specific gravity
- Coefficient of thermal expansion
- Density
- Specific resistance
- Solidus/liquidus temperature range
- Young's modulus of elasticity
- Thermal conductivity
- Impact strength
- Tensile strength and elongation at various temperatures

The handbook includes good narrative summaries of each group of steels and a detailed listing of applicable foreign and domestic specifications. Specification listings for each group of plain carbon steels contain a nominal analysis, the supplier, the condition, and remarks indicating the use and/or hardness, tensile strength, elongation, and proof strength of the group. References 2, 5, and 6 contain general and specific information on classification and specification of plain carbon steels.

#### **10.2.2.2.3 High-strength steel**

High-strength, medium-carbon steels with two or more alloying elements (such as chromium, nickel, molybdenum, tungsten, and vanadium) are a wear-resistant, high-strength choice for springs, studs, bolts, nuts, and all forms of stressed engineering applications. These "high-strength low-alloy"

(HSLA) steels, such as AISI 4340 (0.4 C, 0.8 Cr, 1.8 Ni, 0.25 Mo), have a carbon content high enough to ensure a minimum strength of about 900 MPa (>130 ksi) when fully tempered. The upper limit of the carbon content for this group of steels is near the eutectic point; therefore, even in the fully hardened condition they will retain some ductility. High-strength steels are fairly expensive materials; because their corrosion resistance in the hardened state is only marginally better than that of plain carbon steel, they must be protected. These steels should be kept dry, oiled, or greased if not permanently protected through carefully controlled plating or other treatment. Hydrogen embrittlement can be a problem for these steels if they are not stress relieved during or after processing.

Most high-strength steels have chromium as an alloying element because it increases hardenability of components with thick sections and slightly increases corrosion resistance. Nickel is frequently added to strengthen the iron matrix while providing increased toughness, low-temperature ductility, and fatigue strength. Molybdenum, another carbide former, acts as a grain refiner that also increases fatigue strength and facilitates hardening without temper brittleness by preventing carbide precipitation. Vanadium and tungsten alloying additions have a similar grain-refining effect on high-strength steels attributed to the formation and distribution of carbides. The choice of alloying elements actually used will depend on whether maximum hardness or ductility is required and may be influenced by cost.

#### **10.2.2.2.4 Aluminum alloy 6061**

Aluminum-magnesium-silicon wrought alloys are a range of heat-treatable aluminum alloys characterized by excellent cold working properties. They are suitable for extrusions and have good corrosion resistance characteristics while possessing good mechanical strength. These alloys show less loss in corrosion resistance per proportional increase in mechanical properties than other heat-treatable aluminum alloys. The alloys should be anodized for structural applications. Aluminum 6061 is a

medium-strength, structural Al-Mg-Si alloy. It is a heat-treatable, wrought alloy used for a majority of extrusions. Small quantities are used for sheet and plate. Common forms of the alloy are available in natural aged (T4 and T451) or artificially aged forms (T6 and T651). (An as-fabricated form, O, is available that has inferior properties when compared with others.) It has good weldability, corrosion resistance, and immunity to stress-corrosion cracking. Balanced amounts of magnesium and silicon (1 wt % magnesium and 0.6 wt % silicon) are added to form a quasi-binary Al-Mg<sub>2</sub>-Si alloy. Copper (0.25%) is added to improve mechanical properties, and chromium (0.2%) is added to offset the adverse effect of the copper on corrosion resistance. Iron, titanium, zinc, and manganese are also present in the alloy.

An oxide layer quickly forms on the surface of the metal. Such layers are generally 3 to 5 nm thick, although no equilibrium is reached. Oxide will continue to form, although at a greatly reduced rate. This oxide forms more rapidly in higher temperatures or with increasing humidity. However, the oxide film is generally stable and protects the metal. Thick oxides with improved surface adhesion are sometimes generated to increase corrosion resistance with a hot acid or alkali bath that may contain chemicals, such as chromates, to yield an even higher corrosion resistance. Even thicker coatings are made with anodic processes. The aluminum is made from the anode in an electrolytic solution, such as a 15% sulfuric acid aqueous solution, and then it is sealed by dipping it in boiling water or chromate solution.

Both the naturally and the artificially aged forms of the alloy are solution treated at 530° C then quenched at room temperature. Mechanical properties of the artificially aged alloy are considerably different from the naturally aged. Tensile strength of the alloy improves by approximately 20% with artificial aging. T6 and T651 alloy are nearly 50% harder, with elongation decreasing by a like amount.

Generally, strain hardening has little effect on heat-treated alloys, although high rates of strain may be used in wire, rod, and tube stock of T6 and T651 alloys to improve strength and surface finish.

Information on aluminum alloys is readily available from the Aluminum Company of America (ALCOA). Specific information on alloy properties and specifications can be found in various volumes of the *Metals Handbook*<sup>[2,5,6]</sup>. Reference 7 contains information on the fabrication and properties of aluminum.

#### **10.2.2.2.5 Insulating board (cellulosic fiber)**

Insulating board is a fibrous-felted panel board made from ligno-cellulosic fibers (usually wood or cane) characterized by an integral bond of interweaving fibers. Other ingredients may be added to provide or improve certain properties such as strength and water resistance, and special coatings may be used to impart resistance to flame spreading. The homogeneous boards have density ranges of 10 to 31 lb/ft<sup>3</sup>, and a maximum moisture content of 10 wt % as shipped from the manufacturer.

In packaging applications, insulating board serves a multifunctional role as a positioning, thermal insulating, and energy-absorbing (cushioning) material. Generally, it would be used between outer and inner containers to protect the inner container from thermal and mechanical damage during normal and hypothetical accident conditions of transport. The lightweight board is a readily available commercial product that can be easily cut or drilled to desired design configuration. Table 10.3 shows typical physical and thermal properties of insulating board.

As the cellulosic insulating board is essentially of organic composition, it will undergo combustion in air with an ignition point of about 450° F. The combustibility may affect design decisions for

**Table 10.3. Typical physical and thermal properties of insulating board**

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<b>Modulus of rupture</b>	<b>40-400 lb/in.<sup>2</sup></b>
<b>Shear strength (parallel to surface)</b>	<b>50-200 lb/in.<sup>2</sup></b>
<b>Tensile strength (perpendicular to surface)</b>	<b>500-800 lb/ft<sup>2</sup></b>
<b>Thermal conductivity (at 75°F mean temperature)</b>	<b>0.38-0.44 BTU-in./h-°F-ft<sup>2</sup></b>
<b>Flame spread index (ASTM E 286)</b>	<b>200</b>

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packaging applications involving hypothetical accident scenarios with specific thermal protection and testing criteria. Design modification or compensation, to protect the inner container thermally from short-term combustion effects, might include increasing the wall thickness of the insulating board, using of fire-retardant coatings on the board surfaces, or both.

#### **10.2.2.2.6 Ceramic fiber materials**

Insulations based on ceramic fibers have been used in many applications, particularly following the energy crisis in the early 1970s. Strictly speaking, Fiberglas<sup>TM</sup> is a ceramic fiber but is usually considered to be a separate generic-type product. Base fiber compositions vary widely, depending on application, from common silicates to pure materials such as alumina and zirconia. Because packaging will ordinarily involve only air atmospheres, insulations based on carbon fibers are not considered in this discussion. The fibers may be partially or totally crystalline as determined by composition and processing.

Ceramic fiber insulation is commercially available in a wide variety of forms such as paper, woven cloth, tape, felt, flock, sheet, batt, and boards in both semirigid and rigid form. A discussion of general characteristics and applications of the various forms is in Ref. 8.

The ASTM C-16 committee activities have been invaluable and vital to insulation technology and have resulted in a degree of uniformity in prescriptions for testing insulations, 12 symposia on insulations since 1952, and commonly accepted specifications and standards. Remember, however, that ASTM specifications generally represent the "lowest common denominator," and manufacturers will often claim to meet or exceed the specification. Reference 8 contains extensive tables of data on chemical, physical,

and thermal properties of all generic insulations, including ceramic fiber, that are in general use in industry.

A fact that must be considered with any insulation, and especially with a fibrous material, is that in most cases the material is not isotropic, and properties may vary by a factor of 10 or more with orientation. For example, a property such as conductivity is usually given in the direction of interest or use (perpendicular to usual fiber orientation) and almost never in parallel.

Because of cost, ceramic fiber insulations are generally used only at high temperatures. However, because of low density and very high surface area, hygroscopicity and moisture absorption must be considered for applications that involve lower temperatures because of property changes. In addition, the insulation may release significant volatiles upon first heating, or after exposure to ambient conditions following first heat.

#### **10.2.2.2.7      Fir plywood**

Plywood is composed of thin sheets of wood (called veneer or plies) bonded with an adhesive. The grain of each ply is set at a 90° angle to the adjacent ply. (This distinguishes plywood from glued laminated timber (glulam) the grains of which run parallel to each other.) The plies range from 1/12 to 7/32 in. thick. On an equal weight basis, plywood is about 1.5 times stronger than steel and is therefore a light-weight, high-strength material.

The cross-grain construction of plywood gives it certain advantages over ordinary lumber. Wood is very anisotropic, having considerably lower tensile strength perpendicular to its grain than parallel to its grain. The strength of plywood is nearly the same in either direction. Plywood normally is composed

of an odd number of plies to keep it balanced around the center ply. The balanced construction allows plywood to be used in sheets that are narrower than ordinary lumber and allows fastening with nails or staples much nearer to the edge. Ordinary lumber will shrink or expand from 3 to 6%, depending on humidity levels, across its grain while experiencing very little longitudinal change because the modulus of elasticity parallel to the grain is about 20 times that across the grain. Also tangential shrinkage is about twice radial shrinkage, which leads to twisting or warping of the wood and an uneven distribution of moisture, which can lead to cracking or splitting at the ends. Because of the strength of the adhesive bond, plywood resists the tendency to expand or contract; the shrinkage of plywood is about 1/10 that of lumber. The balanced cross-ply construction reduces warpage, and the cross-grain construction reduces splitting at the edges. Any deviation from the 90° angle of the adjacent plies diminishes the ability of plywood to restrain dimensional change. Another advantage is that plywood can be made in sheets much larger than boards sawed from logs.

Plywood comes in a number of grades. The Department of Commerce, National Bureau of Standards, issued U.S. Product Standard PS 1-83, which has been adopted by the American Plywood Association (APA), formerly the Douglas Fir Plywood Association. The standard defines five groups of trees, classified according to the stiffness of the lumber, used in the production of plywood. Before the PSI standard was issued, specifications were contained in the Commercial Standards (CSs) of the Department of Commerce. The CSs were written in parts specific to four trees, one of which was the Douglas fir, the softwood most commonly used in plywood production. Under the PSI standard, Group 1, the highest stiffness grade, includes Douglas fir grown in northwestern regions, Southern pine, and many others. Douglas fir grown in southwestern regions are in Group 2.

The standard covers appearance grades as well as engineering grades. The appearance of the plywood is ranked N if the face veneer is cut only from heart-wood or sapwood that is free of open

defects. N-graded plywood can receive a natural finish. Other veneer grades range from A through D, wherein the number of allowable defects such as knot holes, patches, or cracks is defined. Only the outside plies (the face veneer) are graded by the standard, and sometimes the two face plies can be of a different grade. Engineering grades are usually C-C or C-D appearance grades and are available as Structural 1, Structural 2, Underlayment, and Plyform grades. For demanding applications requiring the strongest material, Structural 1 material, which can be made from only Group 1 trees, is recommended.

The standard also defines exposure durability grades, depending on whether the adhesive used between the plies can withstand continuous exposure to moisture without delamination. The adhesive used in exterior grades is usually phenol formaldehyde, which leaves a reddish-brown stain along the bondline but can produce bonds that are more durable than the untreated wood itself. Melamine formaldehyde is more expensive but can be used if staining is undesirable. Because wood is susceptible to environmental decay, the faces of exterior plywood should be stained, coated, or painted to prevent ultraviolet deterioration. For long-term exterior exposure, pressure-treated plywood works even better. Exposure 1 plywood (CDX) is made using exterior adhesives, but the faces are less rugged than in exterior grades unless they have been pressure treated. Exposure 2 plywood is bonded with phenol formaldehyde, but the faces are so delicate that it should be used only for interior applications. Exterior and Exposure 1 and 2 plywood can be used in place of vapor barriers if joints are sealed with glue or a vapor barrier tape. Interior grades are bonded with urea formaldehyde, which is inexpensive but strong and tough. Bond strength will be maintained under occasional exposure to moisture, but prolonged exposure will deteriorate the bond.

Plywood carries an APA stamp that shows its exposure durability, appearance grade, engineering grade, and other information. The thickness of the panel may also be listed. Plywood is usually

available in thicknesses ranging from 1/4 to 3/4 in., and the boards are usually 4 by 8 ft. Structural 1 C-C exterior plywood comes in thicknesses of 5/16, 3/8, 1/2, 5/8, 3/4 in. The stamp may also list a span rating which indicates the maximum recommended spacing between supports when the plywood is used as roof decking or subflooring.

For design purposes, plywood is considered to be homogeneous, orthotropic plates. Mathematical formulas have been developed to compute stiffness and strength, stress at proportional or ultimate limits, or estimates of working stresses. The formulas use section properties, which depend mainly on the characteristics of the face plies and the direction of the grain relative to the direction of the stress. The section properties, are independent of the number of plies in the panel. The section properties have been determined by the U.S. Forest Products Service and are called out in the PS1 standard. The APA's "Plywood Design Specification" tabulates section properties and recommended design stresses and methods for the large number of possible combinations of grades and panel thickness (American Plywood Association, P.O. Box 11700, Tacoma, WA 98411-0700, (206)-565-6600). There is a large number of publications about plywood, including Refs. 9 and 10.

#### **10.2.2.2.8 Redwood**

Redwood is an American softwood tree that typically is moderately light, strong, stiff, and hard. It is easy to work, generally straight grained, and shrinks or swells comparatively little. The heartwood resists decay, making it useful for a number of outdoor applications, such as fence material, outdoor furniture, or long-term storage applications.

In general, redwood properties can vary from tree to tree within a species and even from piece to piece by as much as 25%. Also, properties are dependent on the direction of the grain. Other

considerations that may cause redwood properties to vary include age of the tree, water content, and type of wood (heartwood or sapwood).

The specific gravity of dry, young-growth redwood is approximately 0.35, which is the density of water. Water content of the wood can range up to 100% of its dry weight, although commercially available wood is usually 12% or less. It is the most dimensionally stable of any common American wood with volumetric changes limited to about 7%. Typical values for mechanical and other properties of wood may be found in Ref. 10.

#### **10.2.2.2.9 Urethane foams**

A broad range of urethane foams are readily available to the package designer and are often a material of choice. Polyurethanes are derived from the addition polymerization of polyols and polyisocyanates. Flexible foams depend on polyester polyols for good elongation and tensile strength properties and polyether polyols for better hydrolysis resistance and resiliency. Further improvement in resiliency can be obtained from polymer polyols of more complex structure. Rigid urethane foams are the general material of choice for insulation. The rigid foams are cross-linked, closed-cell polymers with a low density. They have good resistance to chemicals and solvents and can be used over a temperature range of 328° to +300° F. Short-term exposure to a temperature of about 480° F does not damage the foams. Their superior insulation properties are derived from the fact that the closed cells entrap the blowing agent, consequently yielding foams with an extremely low coefficient of thermal conductivity. Other important characteristics include good low- and high-temperature dimensional stability; low permeability to water vapor and moisture; and good compressive, tensile, bending, and shear strengths.

The development of polyisocyanurate (FIR) foams provide rigid foams with improved flame retardance. A recent development is the continuous, one-step production of metal panels with integral foam insulation that offers good insulation properties, high strength at low density, and easy installation.

Some properties to consider in the selection of a flexible foam are:

- Density
- Tensile strength
- Tear strength
- Elongation
- Flexural fatigue
- Flammability
- Solvent resistance
- Load deflection and indentation
- Rebound and falling ball
- Humidity aging

It is quite difficult to formulate as many as ten requirements into a given foam and still hold it within the desired tolerances; especially if cost is a major factor. Most often, the formulator can give the end user very close tolerances on the four or five most important properties and reasonably close proximities on the next two to three properties. After these, it is most difficult to come even close to meeting the remaining requirements of the properties desired; therefore, it is important that the end user consider carefully the most stringent requirements and present required properties of major importance in the first four to six listings.

A great number of testing methods have been set up for flexible urethane foams. Some of these testing methods give fairly accurate data on how the foam will perform in its intended environment while other tests provide data that are much less accurate. Among the helpful tests that give reasonably accurate data are the ASTM D 2406 series, which includes the following tests: steam autoclave, compression-set, load-deflection (indentation), compression load-deflection, dry-heat, fatigue, density, tear resistance, and tensile strength. ASTM 1564 is also useful in tear resistance and for the bouncing ball tests.

The British Standard tests are quite useful in a number of categories (BS 3667 for indentation and BS 3379 for tensile strength, elongation, and compression set). British Standard has its own methods of testing for fire resistance and other important properties.

The Society of Plastics Industry (SPI) has a useful grading system for flexible foams, and a number of test methods for specific testing work. Most of these tests are aimed at obtaining data that are directly applicable to environmental conditions.

The Bureau of Naval Weapons has its own standards for testing flexible foams. Its WS-2722 specifications set forth very rigid parameters for foam. A formulator has a difficult time meeting all the specifications. WS-2730 sets forth specifications for a slightly different type of foam.

Rigid urethane foams are far superior to any other polymeric foam in thermal insulation properties. Values are sometimes misleading, but the K-factor of these foams surpasses that of any known insulating material by at least 20% and more. This fact allows lesser quantities to be used or better insulation properties for the same thicknesses of material.

A number of clear-cut methods are available for testing the properties of rigid urethane foams, including ASTM, SPI, and various governmental agencies that have testing methods and specifications. Table 10.4 gives ASTM specifications for testing these foams.

There are many variations of such tests by ASTM and others involved in the industry. SPI has many excellent test methods. Various governmental agencies have tests that more closely meet their requirements as to properties. All such tests are good, and the testing methods used will depend to some extent on the end use of the foam, the density, and various other factors. For instance, one test method may be better for a 2-lb-density foam whereas another method may do a better job on a 12-lb-density foam.

Testing for K-factor is the most controversial of all the testing methods. Manufacturers of foam systems sometimes make claims of K-factors by their private testing methods. These will seldom coincide with the K-factor if done by strict ASTM specifications. The manufacturer usually gives the original K-factor reading on the foam immediately after curing. This figure begins to rise immediately, as some of the gas in the cells begins to permeate out, and is replaced by air. A foam with a K-factor reading of 0.13 immediately after curing may well have a factor of 0.19 at the end of 6 mo. After the 6 mo, the K-factor changes little at normal temperatures and normal conditions.

A good 2-lb-density foam blown with water will have an initial K-factor of about 0.16, which will increase to about 0.21 after 6 mo. The same foam, blown with a fluorocarbon, will have an initial factor of about 0.14 and will increase to about 0.18 after 6 mo. These foams would be considered good representatives for thermal insulation.

**Table 10.4. ASTM specifications for testing rigid urethane foams**

Property	Specification
Density	ASTM 1564-58T
K-factor (Btu/h-sq ft <sup>2</sup> /°F/in.)	ASTM C 177
Water absorption	ASTM C 272-51T
Coefficient of thermal expansion	ASTM D 696
Compression load	ASTM D 1621-59T
Flexural strength	ASTM D 790-59T
Flammability	ASTM D 635
Deflection temperature	ASTM D 648
Dielectric strength	ASTM D 149
Dielectric constant	ASTM D 150
Dissipation factor	ASTM D 150
Volume resistivity (ohm/cm)	ASTM D 257
Arc resistance	ASTM D 495
Tensile strength (parallel and perpendicular)	ASTM C 297-55
Tensile strain (parallel and perpendicular)	ASTM 297-55
Tensile elastic modulus (parallel and perpendicular)	ASTM C 297-55
Adhesive strength	ASTM C 297-55

A sample is weighed, immersed in water for 24 hours, and then weighed again. The volume of water is equal to the weight of the water absorbed divided by the density of water. The percentage of open cells is equal to the volume of water absorbed divided by the volume of the original sample.

**Cell count** The cell count is the number of cells per linear inch.

**Cell structure** This is a visual inspection under various magnifications, usually 10X to 20X.

Water absorption is another test that causes some controversy in the industry. Many systems manufacturers will have testing methods of their own that show more favorable results than is actually the case. A 2-lb-density foam that will pick up under 3% water by volume in a 7-d test and less than 7% in a 180-d test is considered to be an excellent foam.

A number of other tests disclose problems for the industry. Water-vapor transmission is one of these tests. At 100% relative humidity, a 2-lb foam that by ASTM Specifications E 96-53T has a water vapor transmission of 13 perms is considered an acceptable foam for most purposes.

Tests must be run on foams in accordance with the requirements of the end product. All the tests listed will be required on very few foam systems. A foam to be used for insulation in a home, for instance, would need tests run only on K-factor, water-vapor transmission, and water absorption for most end uses. If a foam were to be used as a structural material, tests would have to be run on compressive, tensile, and shear strengths along with the other properties mentioned earlier.

Tests should never be run on a single sample batch. A minimum of three batches of a given formulation should be poured. Samples from these three batches should be tested for the required properties and averages taken. In this manner, statistically accurate values may be obtained. There will always be variations from mix to mix, particularly if the sample batches are hand mixed. This variation is caused by many air bubbles being mixed into the batch. Whenever possible, sample batches should be run with the same proportioning and mixing equipment that will be used in the final process, thus giving much closer values for the manufactured foam.

#### 10.2.2.2.10 Silicone foam

Silicone foams are generally composed of silicon-oxygen compounds that are considered similar in nature to an organic polymer, except that silicon atoms are substituted for the carbon atoms. Silicone foams have several advantageous properties such as their nonburning quality, good electrical insulation, low water absorption, and excellent thermal stability. Silicone foams are available from companies such as Dow Corning Corporation, Sponge Rubber Products Co. (foamed silicone rubber), General Electric Corporation, and McGhan-Nusil.

Three types of silicone foams exist; premixed powders, room-temperature curing resins, and elastomeric foams. The oldest type is the premixed powders, which consist of a polysiloxane resin blowing agent and fillers. This mixture is heated above 160°C, causing the resin to liquefy and the blowing agent to decompose. Nitrogen gas expands the resin, and amines that are released act as catalysts for condensation of the resin. Thus, the expansion and gelation are synchronized so that the resin gels at maximum expansion.

There are three types of powders: Type A, which can be foamed to densities ranging from 0.16 to 0.22 g/cc; Type B from 0.19 to 0.26 g/cc; and Type C from 0.22 to 0.29 g/cc. Types A and B can be foamed in place and are more resistant to thermal shock than is Type C. Type C can be foamed only as a block or sheet and is stronger than Type A or B. Type C also retains more compressive strength than Type A or B at high temperature. Samples of Type C foam have been cycled repeatedly between room temperature and 315°C without cracking and have withstood temperatures of up to 370°C for 72 h with only slight dimensional changes. Type B foam will retain a considerable amount of its compressive strength at elevated temperatures, especially if it is postcured for 48 h at 250°C. These foams should be exposed to the expansion temperatures for at least 4 h to minimize shrinkage. If one is postcuring,

the temperature should be raised by increments of 28°C to the required temperature. Rigid foams are produced by this method.

The room-temperature cure foams are generally a two-liquid silicone component system in the presence of a catalyst producing either flexible or rigid foams. The reaction is slightly exothermic, but temperatures seldom exceed 62°C. Hydrogen gas is liberated as the expanding agent in these foams. Generally, these foams are blended in a high-speed mixer for 30 s and poured. They expand to 7 to 10 times their initial volume. The reaction is complete in 15 min, but the foam remains soft for about 2 h. The foam is hard enough to cut and handle after approximately 10 h with maximum strength obtained in 24 h. Continuous service temperatures of these foams range as high as 350°C.

Elastomeric foams are lightweight, rubbery foams that are also made by mixing two components. The mixing requires about 30 sec and the materials must be poured immediately because expansion occurs immediately upon blending. After approximately 5 min, the foam has developed 80% of its ultimate strength. Maximum strength is generally obtained after 24 h.

Properties of silicone foams depend on both the density of the foam and whether the cell structure is open or closed. Densities of flexible silicone foams can range from 0.11 to 0.54 g/cc. Tensile strengths of these foams are generally low, in the range of 0.1 to 1 MPa; however, low compression sets of less than 1%, chemical inertness, high electrical resistivity, good insulation value, and self-extinguishing characteristics make them an excellent cushioning material. Continuous service temperatures are typically 250° to 350°C for the commercially available materials. Physical property data for some of these foams can be found in Refs. 11 and 12. Vendor literature provides the best source of property data because commercial offerings vary significantly.

#### 10.2.2.2.11 Fluoroelastomers

Fluoroelastomer is the name applied to the broad class of fluorine-containing rubbers originally developed as fluid-resisting elastomers that can be used over a wide temperature range. Copolymers of vinylidene fluoride (VDF) have dominated the market for more than 40 years; however, a number of other materials have been introduced on a smaller scale, including the nitroso rubbers, the phosphonitrilic fluoroelastomers, the perfluoroalkyl vinyl ether copolymers, and the fluorosilicones. Improvements to fluoroelastomers continue to be made in processability, resistance to compression set, low-temperature flexibility, and chemical resistance while maintaining their exceptionally good resistance to embrittlement during long-term heat aging at 200°C. Applications of fluoroelastomers are typical of other elastomers, including belting; caulks; coatings; sealants; low-density products; gloves; tapes; wire and cable jackets; linings for tanks, hoses, pumps and valves; tubing; and gaskets and O-rings.

The primary fluorocarbon elastomers based on vinylidene fluoride are copolymerized with a variety of comonomers, including hexafluoropropylene, 1-hydropentafluoropropylene, tetrafluoroethylene, and perfluoromethyl vinyl ether. Standard nomenclature established by ASTM D 1418 designates these materials as FKM rubbers. These types of materials are generally superior in heat and fluid resistance to the other VDF copolymers. Commercial materials of this type are produced by Du Pont (Viton™), 3M (Fluorel™), Daikin Kogyo (Daiel™), and Montectini (Tecnoflon™). Copolymers with chlorotrifluoroethylene, manufactured by 3M under the trade name Kel-F™, although inferior to the nonchlorine-containing polymers in general fluid resistance, have superior resistance to oxidizing acids such as fuming nitric acid.

The elastomers based on VDF are saturated and cannot be vulcanized with sulfur. As with other saturated rubbers, crosslinking can be brought about by radiation, peroxides, and certain diamines. The

original amine curatives of interest were hexamethylene diamine carbamate (Diak No. 1), ethylenediamine carbamate (Diak No. 2), and N,N-dicinnamylidene-1,6-hexanediamine (Diak No. 3). The amine crosslinking appears to occur by elimination of hydrogen halide to generate unsaturation and crosslinking at the double bond. Hydrogen fluoride adversely affects the heat-aging properties of the cured elastomers and must be removed by a base such as magnesium oxide. The water formed in this process must be removed by postcuring to avoid loss of properties as a result of porosity, splitting in thick sections, or devulcanization reaction. An environment of hot water or steam can have a severe effect on amine-cured fluoroelastomers. The amine-cured fluoroelastomers also suffer from a relatively high degree of compression set at elevated temperatures. Examples of these systems are the Vitons A and B, and the Fluorels 2145, 2175, and 2178. Compression set values (70 h at 200°C) range from 20 to 75%.

The largest volume VDF products now use "incorporated cure systems" (e.g., du Pont "C" curing systems) that do not require the addition of the amine curatives. These products are sold preblended with a quaternary salt cure accelerator (e.g., triphenylbenzylphosphonium chloride); and a diphenol crosslinking agent (e.g., hexafluoroisopropylidene diphenol). Advantages of the incorporated cure systems over the diamine formulations are in processability (better dispersion of curing agent), greater bin stability, better scorch safety, faster curing, and far superior resistance to compression set. As with the amine systems, acid acceptors are used to contain generated hydrogen fluoride. Typically, these are blends of high-activity magnesia and reagent-grade calcium hydroxide. Consequently, water is generated during the cures and must be dealt with as with the amine systems. Examples of this type of material are the Vitons B-910, E-60C, and E430 and the Fluorels 2170, 2174, 2179, and 2181 and Fluorel FT 2350. Compression set values (70 h at 200°C) range from approximately 10 to 30%.

To diminish the effects of water, peroxide-curable VDF copolymers were developed. For this purpose, the polymer was modified to incorporate a cure site of enhanced receptivity by aliphatic radicals. The curing system consists of a peroxide, a co-agent, and an acid acceptor as promoter. The usual peroxides include 2,5-dimethyl-2,5-bis(t-butyl peroxy) hexane (Luperco 101 XL or Varox) and the hexyne analog (Luperco 130 XL). Typical coagents are triallylisocyanurate and Diak No. 7 (an organic triazine). In addition to the common acid acceptors magnesia and calcium hydroxide, sublimed litharge (yellow lead oxide) is used for improved resistance to hot water, steam, and aqueous acids. Improvements should also be realized in thick section cures. This class is represented by the Viton G series of fluoropolymers.

Fillers are usually incorporated into the fluoropolymers to obtain optimum properties. A variety of black and mineral fillers can be used. Filler loadings are normally less than those used for other elastomers, because of the marked increase in cured viscosity with the addition of only small amounts of filler. Typical black fillers are MT Carbon Black, which is used for most applications, and Austin Black, which produces improved long-term compression set resistance at 232° to 260°C. Levels of 30 g/100 g of gumstock are typical. Mineral fillers such as precipitated barium sulfate, fibrous calcium silicate, titanium dioxide, fibrous magnesium silicate, and diatomaceous earth can be used where a nonblack filler or special properties are desired.

Most VDF fluoroelastomers can be formulated to desirable properties without sacrificing processability. Gum viscosity is of primary importance to the determination of processability because this factor affects vulcanizate properties, especially compression set. Formulation viscosity depends on both gum viscosity and filler selection. Formulation stability and safety must also be considered when determining processability because they are strongly affected by compounding ingredients and cure systems. The most workable formulations are compounded using the incorporated cure systems. These rubbers offer the processor the best starting point for maximum processability.

Fluoroelastomer compounding is simple compared with other elastomers, given the few ingredients required. Compounds based on 100 parts by weight rubber gum stock (which may include curative) usually consist of the inorganic base, 6 to 20 parts; filler (reinforcing or nonreinforcing), 0 to 60 parts; accelerators (or curatives if not included in the base), 0 to 6 parts; and process aids, 0 to 2 parts. The selection of the appropriate ingredients is critical to obtain the excellent properties inherent in the gumstock. Formulations are compounded for one of two general uses, O-rings or molded goods. In O-ring applications, the primary consideration is resistance to compression set. In molded goods, the most important physical property in the final vulcanizate is usually elongation at break, with compression set being a minor factor. Compound flow is also important because complex shapes are often required. Proper selection of gumstock molecular weight, cross-link density, initial viscosity, and cure system, fillers, and cure conditions is crucial to achieving the desired properties.

Fluoroelastomer formulations are processable by any standard technique. Open-mill mixing is frequently used except for very low-viscosity gums, which tend to stick to the rolls, and very high-viscosity gums, which are excessively tough. Internal (Banbury) mixing is used where open-mill mixing is impractical, especially for the incorporated cure VDF fluoroelastomers. After mixing, stocks should be cooled as rapidly as possible. Mixed stock to be held more than seven days should be cooled to less than 18° C. Cooled stocks should be allowed to warm up and should be freed of surface moisture before subsequent processing.

Processing of the mixed compounds can involve extrusion preforming; calendaring; and compression, transfer, and injection molding. All types of molding may be carried out at 150° to 200° C. Standard industry molding temperature is 177° C, which allows molding times of 10 min or less for most parts. Special accelerators are available to allow molding at 150° C. Postcuring at 230° to 260° C for 16 to 24 h develops maximum tensile properties and compression set resistance. Good adhesion between

VDF compounds and metals can be obtained during molding and curing if the metal surface is properly prepared and a suitable primer is used.

Properties for the VDF fluoroelastomers vary widely with the formulation. Tensile strengths range from 1000 to 2000 lb/in.<sup>2</sup>, elongation at break 100 to 400%, Shore A hardness 40 to 95, and compression set (70 h at 200°C) 10 to 70%. Service temperatures as low as -40°C are possible with some formulations. Continuous service limits ranging from 3 years at 204°C to 48 h at 316°C have been quoted. Ventilation to remove trace amounts of hydrogen fluoride liberated above 275°C has been recommended.

Attempts to improve the stability of the fluoroelastomers by developing polymers with no C-H groups led to the development of the Du Pont Kalrez<sup>TM</sup> fluoroelastomers—copolymers of tetrafluoroethylene with 40 mol percent perfluoro(methyl vinyl ether) plus a third monomer based on fluorovinyl ether to provide a site for crosslinking. They are designated FFKM by ASTM D 1418. Vulcanization may be brought about by amines such as tetraethylenepentamine and hexamethylenediamine carbamate. Magnesium oxide is present as an acid acceptor for hydrogen fluoride. As with the VDF polymers, a long cure process is required to expel the generated water. Crosslinking agents may include the dipotassium salts of hydroquinone, bisphenols A and AF used in conjunction with an accelerator such as polyethylene glycol.

Another crosslinker mentioned in the patent literature is tetraphenyltin. The chemical resistance of these polymers is outstanding. In particular, vulcanizates can withstand air oxidation to 315°C and exhibit extremely low volume swell in a wide range of solvents. Both of these characteristics are unmatched in any other commercial fluoroelastomer. A few grades are available but not a great variety in properties is not available. Hardness ranges from 70 to 95; tensile strength from 1900 to 2660 lb/in.<sup>2</sup>,

elongation at break, 60 to 170%; and compression set (70 h at 204 ° C), 36 to 50%. These materials are offered only as cured products; gumstocks are not available.

The nitrosofluoro elastomers are represented primarily by the terpolymer of trifluoronitrosomethane, perfluoro(nitrosobutyric acid), and tetrafluoroethylene. The ASTM D 1418 designation for these materials is AFMU. Interest in these materials is due to the non-flammable nature of the compound, even in pure oxygen; excellent chemical resistance;  $T_g$  as low as -51 ° C; and a very low solubility parameter ( $5.2 \text{ cal}^{1/2}\text{cm}^{3/2}$ ). Technical development has been retarded by the somewhat poor heat resistance for a fluoroelastomer, with decomposition evident above 150 ° C; some sensitivity to degradation by organic bases; crosslinking difficulties leading to poor strengths; toxic degradation products; and very high costs (\$300 to \$400 per lb in 1969). Despite these restrictions, the polymer has been available on a developmental scale from Thiokol, primarily with applications in the space program and in protective clothing because of its nonflammability and chemical resistance.

The phosphonitrilic fluoroelastomers are generally derived from the reaction of poly(dichlorophosphazene) with fluorinated sodium alkoxides. A multitude of different polymers can be prepared by varying the alkoxide; the degree of stability depending on the alkyl group. Representative of this class is poly(trifluoroethoxyheptafluoro-butoxyphosphazene) [37002-15-6] (ASTM designation FZ) which reportedly has a very high molecular weight and a very low  $T_g$  of minus 77 ° C. The good flexibility at very low temperature makes this material most attractive in comparison with other oil-resisting polymers, regardless of whether it is a fluoroelastomer type. These materials are offered by Firestone Tire and Rubber Company under the trade name PNF rubbers.

Vulcanization of the FZ rubbers can be brought about by peroxides, sulfur systems, or high-energy electron radiation. The sulfur-accelerator systems give higher tensile strengths (to 2500 lb/in.<sup>2</sup>)

than the peroxide-cured compounds but inferior heat aging and compression set. A basic formulation consists of 100 parts by weight of gum, 30 parts reinforcing filler, 6 parts magnesium oxide acid acceptor, 2 parts stabilizer (such as zinc 8-quinolate), and a curing agent. Mixing and processing are similar to other elastomers. Cures can be as low as 30 min at 160°C plus a postcure of 24 h at 100°C. A wide variety of mechanical properties can be obtained. Hardness ranges from 21 to 88 Shore A, tensile strengths from 260 to 2500 lb/in.<sup>2</sup>, elongation at break 109 to 310%, and compression set (70 h at 135°C) 20 to 89%.

The PNF rubbers show excellent resistance to ozone, solvents, oils, and acids and have excellent hydrolytic stability. These rubbers do not burn, even in oxidizing atmospheres. With properties virtually unchanged from 75° to 120°C with moderate resistance to aging at 232°C, the PNF rubbers are attractive options to the VDF elastomers, except for prolonged high-temperature applications. The FZ rubbers are tougher than the fluorosilicones, which are the only other significant fluoroelastomers to function below -40°C. Applications are in the aerospace, aircraft, automotive, industrial, and oil exploration industries. PNF rubbers were priced at \$40 per lb in 1971.

The fluorosilicone elastomers (designated FVMQ by ASTM D 1418) were developed in the late 1950s at Dow Corning. They maintain the low-temperature flexibility of silicone elastomers but with improved fluid resistance through the incorporation of fluorine into the molecule. The major FVMQ rubbers contain repeating units of methyl 3,3,3-trifluoropropyl siloxane and small amounts of methyl vinyl siloxane to allow cross-linking. The amount of fluorine that can be incorporated (30 to 40 wt %) is limited by the tendency of  $\alpha$  or  $\beta$ -fluoroalkyl substituents to undergo thermal cleavage. Nevertheless, the commercial  $\gamma$ -fluorinated polymers have very good resistance to hydrocarbon fluids.

FVMQ rubbers are compounded with fumed and precipitated fillers; hydroxy-containing, low-viscosity silicone oils; and an aliphatic or aromatic peroxide as a vulcanizing agent. Conventional mixing and molding techniques already described are used to process the fluorosilicones. Typical cure cycles are 5 to 10 min at 116° to 171° C, depending on the peroxide used. As with the other fluoroelastomers, a postcure is recommended to maximize long-term thermal aging properties; 4 to 24 hours at 149° to 204° C is the usual range. During vulcanization, the only significant volatile species formed are by-products of the peroxides. No fluorine-containing species are generated.

Beyond the excellent resistance to fuels, oils, and solvents, the most outstanding feature of the FVMQ polymers are their outstanding low-temperature properties. Glass transition temperatures of 68°C have been reported. The materials are serviceable from 68° to 175°C under immersion conditions and up to 225 °C in dry heat, indicating a very broad useful temperature range. The high-temperature range can be increased significantly by the addition of 0.5 to 3 parts iron oxide per hundred parts rubber. The fluid, compression set, and temperature resistance are not as good as the VDF fluoroelastomers. However, the bulk properties do not show a large change with temperature, giving a fairly flat response on property-temperature curves. Typical property ranges are hardness, 40 to 80 Shore A; tensile strength, 650 to 1400 lb per in.<sup>2</sup>; elongation, 100 to 530%; and, compression set (22 h at 177°C), 11 to 57%.

The fluorosilicones are blendable with conventional silicone rubbers to give intermediate fluid resistance at intermediate costs. They can also be blended with the lower hardness VDF fluoroelastomers to achieve intermediate properties. They can be pigmented to almost any color desired. The FVMQ materials are not recommended for use with ketones and phosphate esters.

The fluorosilicones are available as gums, compounding bases, and uncatalyzed, fully compounded stocks. Also available are liquid heat-vulcanized or room-temperature-vulcanized (RTV) versions. The commercial materials are offered by Dow Corning (Silastic LS series) and General Electric (FSE and FSL).

Over the years, many fluoroelastomers have been prepared in addition to the materials described in this section, including fluorinated acrylates, polynrethanes, polyepoxides, hexafluoroacetone/propylene oxide copolymers, polyfluorals, poly(thiocarbonyl fluoride) (PTCF), and the perfluoroalkylenetriazine polymers (PFAT). They are not generally commercially important for several reasons. Some have been replaced by better materials. Others were thermally unstable in spite of significant fluorine content. PTCF rubbers are interesting because of their good short-term resistance to boiling, fuming nitric acid and boiling 10 percent aqueous sodium hydroxide. The PFAT rubbers have thermal oxidative stability superior to any other fluoroelastomer and excellent resistance to acids and oxidants. On the other hand, commercial development has been hampered by a hydrolytic instability and incompatibility with curing agents and reinforcing fillers. The PFAT rubbers have been produced on an experimental scale for the U.S. government by Hooker Chemical and Dow Corning. Although these materials will probably remain only curiosities or very specialized rubbers, following any developments in these areas would be appropriate.

Many good resources for information on fluoroelastomers include Refs. 12-14. Product literature from the vendors is especially useful.

#### 10.2.2.2.12 Neoprene elastomer

Neoprene (CR) is the generic name used by E. I. DuPont de Nemours Company for polymers of chloroprene (2 chloro-1,3,-butadiene). Companies such as Bayer and Denka also manufacture this type of elastomeric compound. Neoprene is a versatile elastomer used in situations where service conditions require a product to withstand a combination of deteriorating conditions (e.g., oil and ozone). The material is noted for its resistance to petroleum products, refrigerants, ozone, sun weathering, immersion conditions in fresh or salt water, burial, abrasion, impact, and damage from flexing or twisting and for its low degree of permanent deformation. Neoprene products include wire and cable jackets, hoses, belting, gaskets, paving seals, window channels, shoes, tires, and wet suits.

Two groups of general-purpose neoprenes exist—G and W types. The G types are interpolymerized with sulfur and contain a (tetramethyl)thiuram disulfide stabilizer, whereas the W types do not. Other differences between the two include raw polymer stability, cure rate, processing characteristics, and some vulcanizate properties. The G types have a molecular weight distribution ranging from 20,000 to 950,000 with the greatest frequency occurring around 100,000, whereas the W types have a much more uniform distribution with the frequency occurring between 180,000 and 200,000. The differences in the processing of these two types of neoprene are not considered relevant here. The G types produce greater tear strength, a little higher resilience and elongation, and better adherence to natural rubber.

The W types are superior in compression set and heat resistance and have lower hardness and modulus for the same amount of loading. As with other elastomeric compounds, neoprene is generally compounded with fillers to enhance properties for specific applications. Fillers such as metallic oxides are added to regulate the scorch and cure rate of the elastomer; antioxidants are added to increase

resistance to natural aging; antioxidants are added to increase resistance to ozone; reinforcing agents such as carbon black, mineral fillers, or clay are added; plasticizers, oils, or release agents may be added to improve processing characteristics; mineral fillers are used to enhance flame resistance; metallic oxides add resistance to water exposure; and fillers are used to retard growth of micro-organisms. Several varieties of special purpose neoprenes are used for adhesives, cements, and coatings or are copolymerized to offer superior properties.

Properties that may be of interest when choosing an elastomeric compound include tensile strength (ASTM D-412), elongation at break (ASTM D-412), hardness (ASTM D-767), resilience (ASTM D-945), recovery from deformation (ASTM D-395), elongation set (ASTM D-412), and resistance to mechanical abuse (ASTM D-624, D-1054, D-813, D-1630). For neoprene, formulations can be made to give tensile properties ranging from 1000 to 2500 lb/in.<sup>2</sup>, elongation at break ranging from 200 to 600%, and hardnesses ranging from 40 to 95 Shore A. Generally, neoprene is rated for continuous service temperatures of up to 80° to 95°C (176° to 203°F). It can be formulated to withstand for short periods of time temperatures ranging from 54° to 149°C (65° to 300°F).

The electrical properties of neoprene are poor in comparison with other insulating materials such as natural, butyl, and silicone rubber. However, they are adequate for use as insulation in low-voltage (600 V) and low-frequency (60 Hz) applications. As with other properties, the electrical properties vary over a wide range, depending on the type of fillers added to the rubber. Because of the presence of chlorine in neoprene, it does not burn as readily as other polymeric materials. Usually, it will extinguish after removal of the external flame source. Good resources for property information include Refs. 15-17 or the DuPont Elastomer Chemicals Department sales office (216-929-2961).

#### **10.2.2.2.13 Ethylene propylene elastomer**

Ethylene propylene monomer (EPM) rubbers are copolyaers of ethylene and propylene that are incapable of forming substantial rigid, crystalline phases and are therefore amorphous and rubbery. Vulcanizates or crosslinked rubbers are produced by incorporating unsaturated comonomers into the EPM, which yields sulfur and peroxide crosslinkable materials called ethylene propylene di-monomer (EPDM). The EPM and EPDM rubbers are almost always used in a compounded and crosslinked EPDM form that has many of the attributes associated with other polyolefins, such as excellent resistance to acids and alkalies, poor resistance to hydrocarbons, good low-temperature mechanical properties, and good electrical properties. (Some caution should be exercised in the use of black rubbers for electrical insulating applications, although the materials are excellent insulators low filler levels, at high loading levels, the commonly used black rubber filler, carbon black, can greatly increase the conductivity of the material.) As a class, the ethylene-propylene rubbers have good compression set characteristics and the best elevated temperature performance of the commodity rubbers.

Ethylene-propylene rubbers find use principally in modestly demanding gasket and hose applications (e.g., automotive radiator hose). Usually, crosslinkable EPDM-type rubbers are used for elevated temperature service and then almost always with modifying fillers. The usual fabrication method for EPM and EPDM is extrusion, such as for hose and strip, while single shapes can be made by compression or transfer molding. Sponge gasket material is also available. The densities of commercially available polyethylene-type polyolefins range from approximately 0.88 to greater than 0.96 g/cc. Polypropylene usually has a density ranging from 0.90 to 0.91 g/cc. The densities of ethylene-propylene copolymers depend on the ratio of the comonomers and the type and proportion of fillers.

#### 10.2.2.2.14 Polyolefin

The polyolefins, as commonly defined in the plastics community, are a class of thermoplastic polymers that have main chains composed entirely of carbon and hydrogen in the form of methylene, -CH<sub>2</sub>-, units. For this review, only those polyolefins composed completely of methylene units such as polyethylene and polypropylene are discussed in detail. These materials comprise the bulk of polyolefin production.

The commodity polyolefins are relatively inexpensive and are available in myriad forms that, through tailored synthesis and compounding, are employed in a remarkably wide range of applications. Properties range from hard and abrasion resistant to soft and pliable controlled by copolymerization reaction temperature and pressure and catalysts.

This review is intended merely as an introduction to the extremely broad topic of polyolefin technology. An excellent source of further information is Ref. 11. For information of a more theoretical nature, the polymer science texts by Rodriguez, Billmeyer, Tadmor, and Gogos and by Kaufman and Falcetta are useful. The *Corrosion-Resistant Materials Handbook*, edited by D. J. De Renzo, is a good compilation of chemical compatibility information. Extensive use has been made of these references in preparing this summary. For data on particular grades of polyolefins, the best sources are the manufacturers.

Depending on the route of synthesis, polyethylenes may be either of the branched or linear varieties. The differences between branched and linear materials are appreciable. Because polypropylene is synthesized similarly to linear polyethylene, no linear/branched distinction is made in this case. Table 10.5 is a tabulation of some important properties of representative classes of polyolefins.

Table 10.5. Some important properties of representative classes of polyolefins

Properties	High density			Low & medium density			Crosslinked
	Polyethylene homopolymer	Ultra high molecular weight	30% glass fiber reinforced	Branched homopolymer	Linear copolymer	Molding grade	
1. Melting temp. °C, T <sub>m</sub> (crystalline)	103-137	125-138	120-140	98-115	122-124		
1.a T <sub>g</sub> (amorphous)				-25			
2. Processing temp. range, °F (C = Compression; T = Transfer; I = Injection; E = Extrusion)	I: 350-500 E: 350-525	C: 400-500	I: 350-600	I: 300-450 E: 250-450	I: 350-500 E: 450-600	C: 240-450 I: 250-300	
3. Mold (linear) shrinkage, in./in.	0.015-0.040	0.040	0.002-0.006	0.015-0.0050	0.020-0.022	0.007-0.090	
4. Tensile strength at break, psi	3200-4500	5600-7000	7500-9000	1200-4550	1900-4000	1600-4600	
5. Elongation at break, %	10-1200	420-525	1.5-2.5	100-650	100-955	10-440	
6. Tensile yield strength, psi	3800-4800	3100-4000		1300-2100	1400-2800		
7. Compressive strength (rupture or yield), psi	2700-3600		6000-7000			2000-5500	
8. Flexural strength, (rupture or yield), psi			11,000-12,000			2000-6500	
9. Tensile modulus, 10 <sup>3</sup> psi	155-158		700-900	25-41	38-75	50-500	
10. Flexural modulus at 73°F, 10 <sup>3</sup> psi	145-225	130-140	700-800	35-48	40-105	70-350	
11. Izod impact, ft.-lb/in. of notch (1/8 in. thick specimen)	0.04-4.0	No break	1.1-1.5	No break	1.0-No Break	1-20	
12. Hardness: Rockwell							
12a. Shore	D66-73			D44-50	D55-56	D55-80	
13. Coef. of linear thermal expansion, 10 <sup>-4</sup> in./in./°C	59-110	130-200	48	100-220		100	
14. Deflection temperature under flexural load at 55 psi, °F	175-196	155-180	260-265	104-112		130-225	
15. Specific gravity	0.952-0.965						
16. Water absorption after 24 hr. (1/8 in. thick specimen), %	<0.01	<0.01	0.02-0.06	<0.01		0.01-0.06	
17. Dielectric strength (1/8 in. thick specimen), short time, v/mil	450-500	710	500-550	450-1000		230-550	

The linear polyethylenes are further subdivided by density and molecular weight into very low-density (VLDPE), low-density (LDPE), high-density (HDPE), high molecular weight-high-density (HMW-HDPE), and ultrahigh-molecular weight (UHMWPE) materials. These varieties span a wide range of chemical, mechanical, thermal, and rheological properties. Occasionally, the literature will make reference to a medium density polyethylene (MDPE) as defined by ASTM D1248 as having a density between 0.926 and 0.94 g/cc, but most manufacturers simply use a classification based on low- and high-density materials.

Considerable overlap exists in the applications of the varieties of the polyolefins, but some regions of density and molecular weight are particularly well suited for certain purposes. The lowest density polyethylene material now commercially available is VLDPE, which covers the density ranging from 0.88 to 0.91. VLDPE is quite flexible and tough. Film, both stretch and shrink, is a prime use of VLDPE as are flexible tubing and squeeze bottles, which take advantage of its excellent flex life. When compounded with polypropylene, the resulting blend has much improved low-temperature impact resistance over neat (unfilled) propylene homopolymer.

Linear low-density polyethylene is defined by a density ranging from 0.91 to 0.925 g/cc and thus has a slightly higher degree of crystallinity than does VLDPE with attendant differences in properties, whereas attributes such as tensile strength are increased and overall the material is stiffer and less flexible.

Branched LDPE is produced in densities ranging from 0.91 to 0.955 g/cc with 40 to 60% crystallinity. The material usually possesses lower tensile strength and other properties than the similar linear polymer and thus has fallen out of favor in many applications.

High-density polyethylene with a density above 0.94 g/cc is a versatile polymer used for a wide variety of applications. This material is characterized by good mechanical and processing properties and is used in film and sheet as well as in large molded articles. The high crystallinity of HDPE makes it well suited for applications where chemical resistance is important. Injection molding is often used to make HDPE into shapes ranging from thin-wall drinking cups to 5-gal pails. Even larger articles are produced by rotational molding where crosslinkable resin is preferred. Hollow shapes in sizes up to 20,000-gal capacity, such as agricultural tanks, are produced this way.

The high-molecular-weight variant of HDPE, HMW-HDPE, is defined by the same density range as HDPE but has an average molecular weight approximately five times that of the corresponding HDPE. The higher-molecular-weight versions of HDPE and HMW-HDPE possess outstanding low-temperature toughness and are well suited for demanding applications at temperatures to -40°F and even lower. High-molecular-weight polyethylenes have been successfully used as mechanical components in cryogenic systems operating at liquid helium temperatures. The high-density resins, in general, have excellent lubricity and impact and abrasion resistance, all of which improve with increasing molecular weight. The extremely high-performance polyethylene fibers now available are produced from standard HMW-HDPE.

At the extreme of molecular weight is UHMWPE, which possesses many outstanding properties but is somewhat limited in application by the difficulty in molding or extruding the material into shapes of any complexity. In addition to the outstanding wear resistance of UHMWPE, the material may be used at higher temperatures than other "melttable" polyolefins. The cryogenic properties of this material are also extraordinary; examples have been reported of UHMWPE pistons operating without significant wear in liquid helium service. Ultrahigh-molecular-weight polyethylene is available mainly as simple shapes or profiles, which are machined or sometimes hot-forged to final shape.

Polypropylene is an easily molded material usually thought of as a somewhat higher performing material than the moldable polyethylenes. The tensile yield strength, stiffness, and thermal properties of polypropylene are equal or superior to those of HDPE and HMW-HDPE and are superior to the properties of polyethylenes with densities similar to that of polypropylene. However, the toughness and low-temperature properties of polypropylene are generally inferior to those of polyethylenes. To overcome partially the lower toughness and cryogenic properties of polypropylene, impact-grade copolymers with ethylene and, as were mentioned earlier, compounds with VLDPE have been developed that improve the toughness of the material and in some cases allow use to  $-40^{\circ}\text{C}$ . Blends of polypropylene with ethylene-propylene rubbers form a useful class of thermoplastic elastomers known as thermoplastic olefins (TPOs). The impact grades have lower density and deform and process at lower temperatures than does the homopolymer.

Density, molecular weight, and molecular weight distribution (MWD) have significant effects on the chemical, mechanical, and rheological properties of the polyolefins. Density is closely related to crystallinity within a class of polyolefins and is commonly specified in g/cc. Mechanical properties, softening temperature, and permeability are strong functions of density. Molecular weight is usually specified in the literature as a unitless number, although (to be rigorous) the units are grams per mole. Typical values range from the tens of thousands to several million. Often, rather than specifying molecular weight, suppliers, especially of molding resins, will provide a melt index number in grains per 10 minutes which is closely related to molecular weight although not necessarily linearly proportional. In general, the lower the melt index, the higher the molecular weight. MWD is usually reported as a polydispersity index that indicates the broadness of the molecular weight distribution. In general, narrow distributions provide high-impact properties while medium to broad distributions improve processability, strength, and creep resistance.

Together, density, molecular weight, and MWD characterize a particular polyolefin; these attributes are employed throughout the industry to specify these materials. Table 10.6 displays the responses of various macroscopic chemical, mechanical, and rheological properties to density, molecular weight, and MWD.

The chemical resistance of polyethylenes and of linear materials in particular is superb. However, polyolefins share a common trait of being attacked by aromatic and chlorinated solvents; the severity of attack decreases markedly with increasing density and molecular weight. The data in Table 10.5 reflect this behavior in an increase of environmental stress crack resistance (a combined chemical and mechanical effect) with increasing density and molecular weight. Whereas LDPE swells appreciably and can show some residual solubility at room temperature, no solvents exist that even significantly swell UHMWPE under the same conditions. Most of the polyolefins are attacked, even to the point of dissolution, in hot aromatics, except for UHMWPE, which undergoes reversible swelling.

Concentrated, strong oxidizing acids attack all of the polyolefins with some degree of severity, depending on the density and molecular weight of the polymer. Weaker solutions of these acids have little effect on most of the polyolefins. In particular ion concentration nitric acid solutions can be used to remove actinide surface contamination without damage to the bulk material. Other oxidizing agents, such as peroxides and ozone, can have long-term deleterious effects. Attack is preferentially at tertiary carbons at the branch points on the polymer chains, so the lower-density polyethylenes and polypropylene would be expected to be the most susceptible to this form of chemical attack. This same susceptibility to preferential oxidation at chain branch points also accounts for the oxidative degradation, which can be a problem in high shear or extended dwell melt processing. As an extreme example of susceptibility to oxidative attack, all polyolefins have high heats of combustion and readily burn in air and sustain combustion.

**Table 10.6. Basic polyolefin parameters and their influence on container properties**

	Density		Molecular weight		Molecular weight distribution	
	Increases	Decreases	Increases	Decreases	Broadens	Narrows
Environmental stress	▼ <sup>a</sup>	▲ <sup>b</sup>	▲	▼	▲	▼
Impact strength	▼	▲	▲	▼	▼	▲
Stiffness	▲	▼	—	—	—	—
Hardness	▲	▼	—	—	—	—
Tensile strength	▲	▼	—	—	—	—
Permeation	▼	▲	—	—	—	—
Warpage	▲	▼	—	—	—	—
Abrasion resistance	—	—	▲	▼	—	—
Flow processability	—	—	▼	▲	—	—
Melt strength	—	—	▲	▼	▲	▼
Melt viscosity	—	—	▲	▼	▼	▲
Copolymer content	▼	▲	—	—	—	—

source: Hoscht  
Celanese

<sup>a</sup> The symbol "▼" indicates a decrease in the item in the left column; e.g., as the density increases the environmental stress decreases.

<sup>b</sup> The symbol "▲" indicates an increase in the item in the left column; e.g., as the density decreases the environmental stress increases.

Ultrahigh-molecular-weight polyethylene is reported to form a protective carbon layer in some instances, which greatly increases its resistance to some aggressive environments. For example, UHMWPE has been successfully used in contact with sulfuric acid at 450°F; a char layer protects the bulk material from degradation.

As a result of the relatively low affinity of polar substances for the nonpolar polyolefin structure and the high degree of crystallinity in many of the grades, the barrier properties of polyolefins are quite good. Water absorption and permeation are particularly low in these materials, the differences between grades being due almost entirely to differences in density. For example, a reported room-temperature moisture-permeability coefficient for 0.922-g/cc polyethylene is  $9 \times 10^{-9}$  cc standard temperature and pressure centimeter second<sup>-1</sup> per centimeter of mercury centimeter<sup>-1</sup> while that for a 0.96-g/cc polyethylene is only 1.2 in the same units. By comparison, the moisture permeability coefficients for 0.907 g/cc polypropylene and ethylene-propylene rubber, in the preceding units, are  $5.1 \times 10^{-9}$  and  $45 \times 10^{-9}$  (at 37.5°C), respectively. Barrier properties for many other gases are also quite good, although polypropylene in particular has relatively high permeability to oxygen. Many nonpolar solvents, particularly aromatic and chlorinated solvents, pass readily through polyolefin films.

Often, for end use or processing, polyolefin resins are compounded with various additives to modify or improve some characteristics. For example, to improve stiffness and reduce cost, glass fibers or mineral fillers are blended with the materials, usually in the range of 10 to 40%, mainly for use as injection molding resins. Fiber-reinforced polyolefin formulations show greatly improved mechanical properties over neat resin, and reinforcement does improve the poor thermal performance of these materials. Vulcanizable polyolefin rubbers and chemically crosslinkable polyethylenes are compounded with crosslinking agents before forming. Various flame retardants are used with polyolefins. Usually, they are the additive type and include polybrominated diphenyloxide, halogenated paraffins, and

ammonium polyphosphates. The mechanism of action of the flame retardants is primarily decomposition to form blanketing nonflammable gasses. Even with flame retardants, the polyolefins are excellent sources of fuel.

Care should be taken when using polyolefins, including ethylene propylene elastomers, as packaging for fissile materials. Polyolefins are excellent neutron moderators; however, compounding with neutron poisons, such as boron, can effectively reduce the risk of accidental criticality. Polypropylene-1 percent boron is commonly used as a neutron shield.

#### **10.2.2.2.15 Silicone elastomers**

O-ring seals made with silicone heat-cured rubbers offer a number of unique properties derived from the silicone molecular backbone rather than carbon-based polymer chemistry. Properties are maintained across a range of temperatures from  $-100^{\circ}$  to  $+315^{\circ}\text{C}$ . The improved properties include weathering and chemical resistance, physiological inertness, lubricity, electrical properties, and compression set resistance.

Heat-curable silicone rubbers refer to compounded polydimethylsiloxane rubber with catalyst and additives (if any) mixed and milled into siloxane polymers. These are extruded or molded and cured for about 10 min in the  $120^{\circ}$  to  $177^{\circ}\text{C}$  temperature range. Applications having service temperatures above  $66^{\circ}\text{C}$  or where dimensional stability is a key parameter require postcuring at the maximum service temperature. A wide range of properties can be achieved by the compounding.

Shore A durometer hardness will vary from 5 to 90. Tensile strengths are 2.1 to 10.3 MPa, and tear strengths are 0.3 to 1.5 MPa with elongations to 1000%. Compression set by ASTM D395, method

B, is as low as 5 to 40% for a specific gravity range of 1.0 to 1.7. Volume expansion coefficient is  $6 \times 10^{-4}$  to  $8 \times 10^{-4}/K$ , and the linear expansion coefficient is  $2 \times 10^{-4}$  to  $3 \times 10^{-4}/K$ .

Thermal conductivities are from 0.22 to 5.9 W/m/K. Silicone rubbers retain their properties to a much greater extent at high temperatures than do most organic rubbers. Silicones with a tensile strength of 10 MPa at 25°C will retain 1.5 MPa at 315°C in oxygen. Performance is even better in the absence of oxygen. In contrast, the organic rubbers are generally useless at 250°C.

While the O-ring seal manufacturers themselves represent the best source of data for the specific compounds used in product line and fillers are often used to modify properties, a summation of the typical properties is available. Low-compression-set silicone rubber has an elongation of 130%, tensile strength of 6 MPa, shore hardness of  $60 \pm 5$ , and a service temperature ranging from -50° to +260°C.

### **10.3 FABRICATION**

The following fabrication methods are preferred other methods may be used, but justification must be documented for special nuclear material packagings:

- Machining
- Forming
- Forging
- Heat treating
- Welding
- Bolting
- Bonding (glue)

- Plating
- Painting

The following sections address the use of the preferred fabrication methods for each of the materials used in special nuclear material packagings. The positive and negative experiences with these fabrication methods are also given.

### **10.3.1 Machining**

#### **10.3.1.1 Austenitic stainless steel**

Austenitic stainless steels are readily machinable by a variety of conventional methods such as drilling, turning, and sawing, but they also have problems. Each has a high work-hardening rate and, because of high ductility, it may be somewhat gummy (stringing, tangled chips) during machining. Work-hardening effects can be minimized by maintaining feed rate above minimum for the material and performing the operation uninterrupted. Sufficient working during machining may introduce slight magnetic character (as a result of a strain-induced phase transformation) into the otherwise nonmagnetic material. A common example is that the threads on some Type 304L bolts are slightly magnetic.

#### **10.3.1.2 Carbon steel**

Machinability of carbon steel is controlled mainly by carbon content, impurities, and hardness. The lower carbon steels are difficult to machine with a good surface finish without the addition of additives such as sulphur, phosphorus, lead, or tellurium. The poor surface finish can be minimized by hardening the material before machining; however, this process is not economical for the lower carbon

steels. With the exception of the specially developed "free machining" steels, the medium carbon steels are probably the most readily machined of the plain carbon group due largely to their hardenability. Typically, these materials are machined by a variety of conventional methods such as turning, milling, drilling, and sawing in the hardened or tempered condition to achieve the best surface finish. High hardness and uniform distribution of oxide inclusions facilitate machining to a good surface finish. The higher carbon steels are more difficult and more expensive to machine. In general, if machining is required, semifinish machining is performed on material in the normalized condition followed by hardening or tempering of the material before machining to final dimensions by grinding. Processing required to achieve hardness can cause distortions that may have to be compensated for in machining.

#### **10.3.1.3 High-Strength Steel**

Many of the materials listed will be too hard to machine after hardening and tempering because they will be above 1170 MPa (>170 ksi) tensile strength. The best condition for machinability is hardened and tempered at 1000 MPa tensile strength, and for some purposes it may be necessary to obtain this condition and re-hardening and tempering before final grinding. Annealing at a temperature of 650° to 670°C for 2 h removes most cold work before machining. For normal purposes, the steels should be rough machined in the normalized and annealed or spherodized condition (depending on composition), which generally results in some tearing, then finally ground after hardening and tempering. Where parts are machined from rough materials in the finally hardened and tempered condition, it may be advisable to stress relieve at the tempering temperature before finish machining. High-speed or tipped tools are essential for cutting the steels listed. Spark erosion and electrochemical machining techniques can be used to shape fully hardened parts, but they will seldom be economical.

#### **10.3.1.4 Aluminum alloy 6061**

Aluminum 6061 is easily machineable by common methods in the heat-treated condition. Because of the presence of hard intermetallics, high-speed or tipped tools should be considered. Some minor problem may occur in chip formation, particularly in T4 and T451, because of their relatively higher ductility. T6 and T651 alloys are generally resistant to strain hardening, which makes them somewhat more easy to machine.

#### **10.3.1.5 Insulating board (cellulosic fiber)**

The insulating boards are readily cut to the desired shape using conventional woodworking tools. Standard wood drill bits, hole saws, and cutting saws (band, radial arm, table, and saber), can be used. Carbide-tipped bits or blades can be used but are not necessary. Fine-tooth saw blades are recommended to minimize tearing of the lightweight, fibrous material.

#### **10.3.1.6 Ceramic fiber materials**

Ceramic fiber materials present no major problems in machining using ordinary carpentry tools (e.g., saws, drills, files, and shears). Care should be taken to minimize dusting because some products might contain respirable fibers.

#### **10.3.1.7 Fir plywood**

Sharp knife techniques will produce less damage to the fir plywood structure than sawing. Knives must be kept sharp and properly aligned. Saws tend to tear the fibers more severely than knives;

however, properly maintained saws can cut the wood fibers quite adequately. When cutting with a table saw, the good side of the wood should face up; with a portable electric saw, the good side should face down. End user sanding of the face veneers should be avoided. Panels with B-grade or better faces are sanded smooth in manufacture to fulfill the requirements of their intended end use. Some grades are only touch sanded for sizing to make the panels more uniform while some grades are left unsanded. End user sanding could lead to damage of the face veneer or to a lack of balance in the panel.

#### **10.3.1.8 Redwood**

The ease of working wood generally varies with the specific gravity of the wood, with the lower specific gravity woods being easier to work. Also, smooth-grain woods are generally easier to work; therefore, redwood is relatively easy to work with common woodworking techniques. However, redwood is also fairly easily to split, so care must be taken, depending on the grain direction and the operation being performed.

#### **10.3.1.9 Urethane foam**

Urethane foams, regardless of density and open or closed pore status, are best machined by dry grinding methods. The dust produced may be treated as a nuisance dust. Hot wire cutting and shaping may be undertaken for simple shapes. Best thermal protection performance can be achieved by a "foamed in place" approach when the application and the exothermic reaction will allow.

#### **10.3.1.10 Silicone foam**

Silicone foams are best machined by dry grinding; however, the very high elongation to failure of the base polymer makes exact dimensions difficult. The softer materials are better foamed to shape. Thermal protection performance is enhanced by “foamed in place” applications, and little, if any, exotherm is evident.

#### **10.3.1.11 Fluoroelastomer**

Machining is not recommended for these materials beyond trimming of molding sprues and flashings. Sharp-bladed instruments should be used.

#### **10.3.1.12 Neoprene elastomer**

Generally, neoprene is formed to specific shapes, as are all other elastomers. In some instances, gasketing material may be stamped or die cut. Machining of elastomeric compounds is generally very difficult if not impossible. The compound would generally be frozen with liquid nitrogen and torn rather than machined.

#### **10.3.1.13 Ethylene propylene elastomer**

The ethylene propylene elastomer is generally molded to shape, because its low modulus and high tear strength do not allow conventional machining operations. The material may be die cut but with some difficulty.

#### **10.3.1.14 Polyolefin**

Formed articles may be further machined to provide features either difficult or uneconomical to form by basic molding techniques. Virtually all of the common machining operations,—such as sawing, turning, planing, milling, drilling, and thread cutting may be used on polyolefins. Sharp carbide tools are recommended with rapid removal of swarf and turnings. Cooling with air or compatible cutting coolants is usually necessary only when deep cuts are used. When dimensional tolerances are important, annealing of the blank is recommended to reduce residual stresses. The suitability of various machining operations may be limited by the lower hardness of the lower-density polyethylenes.

#### **10.3.1.15 Silicone Elastomer**

Because of their properties, silicones are almost impossible to machine or shear. Cutting operations are effective. Silicone products are generally molded to final shape.

### **10.3.2 Forming**

#### **10.3.2.1 Austenitic stainless steel**

Cold working (plastic deformation below recrystallization temperature) is a common forming method for stainless steel. In the initial stages of cold work, austenitic stainless steels are readily formable, but as the degree of work increases, work hardening (and therefore the power requirement) increases. Depending upon the degree of work required, stress relief or solution treatment may be required following cold working. Hot working involves plastic deformation at temperatures above recrystallization temperature. Hot working requires less power and typically presents few cracking

problems as a result of work hardening, but it is more difficult to control in terms of potential oxide contamination of surfaces and precise dimensions of finished components. These materials are readily suited to bending, rolling, drawing, extruding, punching, and other forming operations with appropriate attention given to temperature, extent of work, and other control variables.

### **10.3.2.2 Carbon steel**

Forging, drawing, extruding, rolling, and other forming processes are standard operations in the fabrication of plain carbon steel.

The deoxidation practice used to make a steel will affect its suitability for forming. One of the cheapest steels suitable for many forming applications is hot-rolled, commercial quality, rimmed steel. For an increase in cost, better surface finish and superior formability are available with cold-rolled drawing quality, special killed, and temper-passed steel. Killed steels are preferred where uniform mechanical properties and severe forming applications are required, although they may be less tolerant to handling damage and may have inferior surfaces. A consideration when using rimmed steels is that they can strain age, especially after working, which can change their subsequent formability characteristics. Artificial aging can be used to prevent this effect without appreciably altering tensile strength; however, yield strength and hardness will increase and elongation will decrease.

### **10.3.2.3 High-strength steel**

These steels cannot be cold worked to any great extent without becoming too brittle for practical use. Some wire specifications are cold drawn and tempered, and some bar alloys have final cold-sizing passes, but in general, full annealing between cold-working operations will rarely be required because

these materials are almost always hot worked and then heat treated to establish mechanical properties. Warm forming may be used to process these steels to protect against loss of mechanical properties already established. Typically, this is accomplished by heating the material close to the temperature at which it was previously tempered and forming immediately.

#### **10.3.2.4 Aluminum alloy 6061**

Aluminum 6061 can be extruded, rolled, shaped, and formed into sheet or plate. Cold working has little effect on the artificially aged T6 and T651 alloy forms, although it can be useful in adding strength to the T4 and T451 alloys. The artificially aged forms may be annealed to improve ductility as desired.

#### **10.3.2.5 Insulating board (cellulosic fiber)**

The rigid boards cannot be easily formed to contour. Normally layers are bonded together and shapes machined from the composite blank.

#### **10.3.2.6 Ceramic fiber material**

Not applicable.

#### **10.3.2.7 Fir plywood**

Not applicable.

#### **10.3.2.8 Redwood**

Redwood stock may be pressure treated or steamed to curved contours, but the practice is not recommended for long-term applications under normal storage conditions.

#### **10.3.2.9 Urethane foam**

Urethane foams may be blown into complex molds or formed in place if required by packaging design. Adhesion to the surrounding mold or container walls can be adjusted by surface preparation and mold release compounds.

#### **10.3.2.10 Silicone foam**

Silicone foams can be easily molded to desired shapes. Mold pressures are low, and the polymer flows well during the foaming step. The cure is rapid with very little shrinkage. Complete cure properties are obtained in less than 24 h. However, care must be taken during the foaming operation to follow the manufacturer's instructions exactly if the foam is to be molded and removed from the mold for further use.

#### **10.3.2.11 Fluoroelastomer**

Fluoroelastomers are usually molded to shape for best properties. Fillers may be incorporated in order to tailor properties.

### **10.3.2.12 Neoprene elastomer**

As with other polymeric materials, neoprene can be molded by compression, transfer, injection, blow, or vacuum and wrapped-mandrel methods. Products such as spark plug boots, radiator hoses, neck tubing, electrical connectors, seals, and O-rings are generally formed this way. Problems that may be encountered with molding neoprenes include shrinkage, poor knitting or flow cracks, air marking, sponging and porosity, entrapped air, distortion, backrinding, tearing on removal, pebbling, flashing, and sticking in the cavity. These are the basic problems of molding any type of polymeric compound, and molders generally know how to address such problems.

Neoprene may also be extruded to form products such as hose, tubing, window gasketing, and wire covering. When extruding neoprene, the normal problems of die design, optimum extrusion rate, and temperature control must be addressed.

Another option for forming neoprene is calendaring. This method is used to produce gaskets, pads, and diaphragms. Again, normal variables such as volume of feed, temperature of rolls and of stock material, and roll pressure must be addressed when calendaring neoprene or other elastomeric materials.

Neoprenes can be dissolved in certain solvents. This technique is generally used to form neoprene adhesives or coatings.

### **10.3.2.13 Ethylene propylene elastomer**

The usual fabrication method for ethylene propylene elastomer is extrusion for continuous product. Compression or transfer molding is appropriate for single shapes. Gaskets and O-ring seals may be either molded solid or as a self-skinning sponge.

### **10.3.2.14 Polyolefins**

Being thermoplastics, low- to medium-molecular-weight polyolefins can be processed by most of the common melt phase polymer fabrication methods such as injection molding, extruding, blow molding, film blowing, rotomolding, solid-phase forming (forging), thermoforming, and foaming. Rheological behavior and thermal stability are the most important properties in determining processing characteristics. The thermal properties of polyolefins are dominated by the relatively low melting point of the materials and susceptibility to thermal degradation and oxidation. Thermal degradation is a primary consideration in the plasticating process where excessive temperatures can develop from shearing work on the melt. Because a plasticating step is included in most melt-phase forming processes (e.g., extrusion and injection molding), opportunities for polymer degradation are usually present. Typically, thermal degradation can be a problem as processing temperatures approach 200°C or if dwell times at lower melt temperatures become excessive. In the case of high molecular-weight polyolefins, the high shear stresses and associated high temperatures sometimes required for processing can lead to mechanically induced chain scission and consequent degradation of properties. While both high molecule weight (HMW) - HDPE and UHMWPE are synthesized as linear polymers, the extreme chain lengths in these materials, which can be from 50 to 100 microns, and the resulting entanglements preclude significant flow even at high temperatures. The higher-molecular-weight versions of HMW-HDPE can be processed with some difficulty by standard polymer processing operations, but UHMWPE must be processed using

metallurgical forming techniques, such as ram extrusion, forging, and compression molding. UHMWPE, while technically displaying a melt transition, does not assume a "liquid" melt state. Reportedly UHMWPE has been used in applications at temperatures of up to 232 °C; the inherent resistance to flow of the material and the formation of a thin carbon layer at exposed surfaces appear to account for this high-temperature survivability.

#### **10.3.2.15 Silicone elastomer**

Silicone elastomers are easily extruded or molded to complex shapes and cure quickly. Their fluidity and low surface tension allow them to be easily formed to complex shapes and contours. Their thermal and dimension stability retain these shapes even when used as a mold for other polymers.

### **10.3.3 Forging**

#### **10.3.3.1 Austenitic stainless steel**

The typical working temperature for forging of Type 304L stainless steel is in the range of 900° to 1200°C. At higher temperatures in this range, the possibility of the presence of ferrite (particularly during initial breakdown of a cast ingot) can invite cracking problems. This problem can be minimized by a solution treatment soak before forging and by lowering forging temperature. In addition, forgings finished below typical solution treatment temperature should be solution treated following forging.

### **10.3.3.2 Carbon steel**

Carbon steels are readily forged into a variety of shapes using hot-, warm-, or cold-forging processes with standard equipment. Only the free-machining steels with additives, such as sulphur, are difficult to forge. In general, forgeability increases with deformation rate. In general, forging temperature decreases with increasing carbon content. Forging is typically performed at temperatures between 1200° and 1300°C. The dimensions (thickness) and complexity of components that can be successfully forged from carbon steels are limited not by the material's forgeability but by the cooling off of the hot workpiece when it contacts cold tooling. Usually, forgings are purchased, by specification, in one of four conditions: heat treated for machinability, heat treated for final mechanical and physical properties, specially heat treated for dimensional stability, or heat treated as-forged.

### **10.3.3.3 High-strength steel**

High-strength steels are readily forged into a wide variety of shapes using hot-forging methods. Warm and cold forging are used to a lesser extent because of the higher strength of these materials (and resistance to deformation) and the need to carefully control intermediate thermal treatment. Generally, hot forgeability improves with deformation rate and hot-forging temperature decreases with increasing carbon and alloying content. Forgings are usually specified in the as-forged or heat-treated condition. Various heat-treated conditions may be called out to indicate that the forging is to be machined after forging or that final properties are to be obtained. Other thermal treatments after forging may be specified to control dimensional distortion, residual stresses, surface hardness, etc.

#### **10.3.3.4 Aluminum alloy 6061**

Special care must be given to forging parts of Aluminum 6061. Generally, only T4 and T6 alloys are available for forging because the other forms use stretching to relieve built-in stresses caused by water quenching. Forgings are held at 175°C for 8 h. Forgings may be more susceptible to cracking because of the chromium content of the alloy.

#### **10.3.3.5 Nonmetallic materials**

Forging-type technologies are generally not applicable to the nonmetallic materials of interest.

### **10.3.4 Heat treating**

#### **10.3.4.1 Austenitic stainless steel**

Heat treatment of Type 304/304L and Type 316/316L grades of stainless steel is typically performed to homogenize the material composition (most commonly to return chromium carbides to solution) or to provide stress relief following welding or some forming operation. Solution treatment (sometimes called annealing) to return chromium carbides to solution is performed by soaking the steel at temperatures from 1050° to 1150°C for 1 h followed by a rapid cool to room temperature (typically a water quench). Typically, temperatures at the low end of this range apply to Type 304/304L steels and temperatures at the high end apply to Type 316/316L steels. Stress-relief heat treatments are typically performed by soaking at temperatures from 500° to 900°C (most common at the higher end of this range) followed by a slow cooling process. Potential problems include inadequate solution treatment or too much time stress relieving (both may generate carbides on grain boundaries), which compromise localized

corrosion resistance in many environments. In addition, embrittlement of welds containing ferrite occurs during prolonged exposure in the temperature range from 400° to 800°C with Type 316/316L alloys being somewhat more susceptible than Type 304/304L alloys.

#### **10.3.4.2 Carbon steel**

Low-carbon steels are commonly used for deep drawing sheet because of their low hardenability and good ductility. They require annealing only after considerable work. Annealing is typically performed in the 900° to 950°C temperature range for about 20 mm/in. of cross section followed by a slow cooldown. Scale will form at these temperatures unless the atmosphere is controlled (a reducing atmosphere is recommended over a neutral one). Normalizing, a similar process, is performed after forging and is recommended before any carburizing. The lower-carbon steels are typically not hardened and tempered because the practice does not warrant sufficient increase in strength or hardness to offset the expense.

Medium-carbon steels require intermediate annealing at temperatures near 900°C (20 min/in.) after any significant amount of cold working. Normalizing (at temperatures around 900°C for 10 min/in. of cross section followed by slowly cooling in still air) is usually performed after forging or other hot-working operation to reduce stresses, homogenize the structure, and increase ductility. Subsequent hardening is accomplished by heating to temperatures around 850° C (again, 10 min/in.) and water or oil quenching. Tempering is required shortly after quenching to avoid cracking or other stress-related problems. Specifics of tempering (time and temperature) will vary greatly, depending on the desired combinations of strength or hardness and ductility. Scale removal may be required by blasting or acid pickling after thermal treatment.

### 10.3.4.3 High-strength steel

It is important that high-strength steels are heated slowly to temperature for all thermal treatments to avoid distortion or cracking, especially where the workpiece has greatly varying section thicknesses. Sub-critical annealing for moderate times at temperatures of 650° to 670°C is used to remove cold work. Spheroidizing is performed on alloys containing appreciable quantities of chromium, vanadium, and tungsten to aid machinability. This cyclic heating process involves heating well into the austenitizing range (800° to 920°C) for 30 min, transferring to a furnace at 670° to 700°C for 2 h of soaking, and then repeating this two-step process one or more times. Spheroidizing creates a microstructure with finely dispersed, round carbides (hard particles) in a soft ferritic matrix. Normalizing (heating to 800° to 920°C for 30 min and cooling in still air) is performed only when spheroidizing is not appropriate or required. The considerable air hardening that occurs during the normalizing treatment is tempered by a subcritical anneal performed at 650° to 670°C.

Intentional, through-thickness hardening is accomplished by heating to 780° to 900°C for 20 min/in. of cross section and quenching in oil or circulating air. Tempering is then performed without delay. A deep freeze treatment (-80° to -100°C) is frequently used either following hardening or between multiple tempering treatments to stabilize the parts dimensionally and maximize hardenability.

Martempering is a special thermal treatment often used to reduce the residual stresses that develop while quenching from high temperature. This process involves heating to the austenitizing range, quenching into a hot medium such as hot oil or molten salt, stabilizing at the quench temperature, and then cooling at a moderate rate to room temperature in air. This process reduces the thermal gradients through the section, thereby reducing residual stresses and distortion. The effect of this benefit can be

to simplify the fixturing normally required to prevent distortion during quenching, which can reduce processing costs.

Austempering is another thermal treatment used for these high-strength steels in place of conventional austenitizing, quenching, and tempering cycles. It is used to maximize ductility or notch toughness at a given hardness and to reduce distortion. If appropriate for use, it can result in considerable time savings over conventional thermal treatments for hardening or tempering. With austempering, the material is austenitized at 790° to 870°C and then quenched to a molten salt bath that is held at a constant temperature slightly above the temperature at which the hard martensitic structure begins forming (typically the bath temperature is between 260° and 400°C) until all the material has transformed isothermally to a structure called bainite. Then the material is cooled to room temperature in still air. No further tempering is necessary.

The specifics of any of these thermal treatments will be governed by the composition of the alloy properties or processing qualities desired. General information for many classes of alloys and specific information on heat treating of selected alloys is available in Ref. 6.

Unless the furnace atmosphere is carefully controlled during heat treatment, some scale will form. Scale can be removed by grit blasting.

#### **10.3.4.4 Aluminum alloy 6061**

Heat treating, generally called aging, is used in the T6 and T651 alloys to increase hardness and tensile properties. It can also be used to relieve residual stresses caused by quenching, although the T451 and T65 1 alloys use stretching to achieve some stress relief. Heat treating forms a finely dispersed

precipitate, which discourages slippage of the plates. Temperatures for aging aluminum 6061 are relatively low, usually between 160° and 175°C. Extrusions and forgings are held at the high end of the range for 8 h, with other product types being held at the lower temperature range for 18 h.

#### **10.3.4.5 Insulation board (cellulosic fiber)**

No advantages in properties or life-cycle performance will result from heat treating insulation board. Heat treating may be used to reduce the inherent moisture content if desired.

#### **10.3.4.6 Ceramic fiber material**

Not applicable.

#### **10.3.4.7 Fir Plywood**

Not applicable unless dried to reduce moisture content in closed containers. Consult reference materials for proper drying temperatures based on construction features.

#### **10.3.4.8 Redwood**

Heat treating or seasoning wood is generally done to control the moisture content, of the wood. Controlled seasoning of the wood reduces warping and shrinkage, increases strength and nail-holding power, decreases susceptibility to attacks from insects and fungus, and increases the ability of the wood to take preservative, fire retardant, or paint. The seasoning can be done by air drying, which does not remove all of the moisture, or by kiln or oven drying. Kilns normally use temperatures between 110°

and 180°F although higher temperatures are often used. Although higher drying temperatures are popular because they reduce drying time, they can decrease strength. Drying time depends on the desired moisture content of the final product.

#### **10.3.4.9 Urethane foam**

No advantages in properties or life cycle result from heat treating urethane foam.

#### **10.3.4.10 Silicone foam**

Postcure at or slightly above extended service temperatures in a circulating air oven will enhance all of the positive aspects and properties of silicone foam.

#### **10.3.4.11 Fluoroelastomer**

Postcure at or slightly above intended service temperatures for two hours will generally result in slightly superior properties for fluoroelastomer.

#### **10.3.4.12 Neoprene elastomer**

No advantage can be gained by postcure heat treatment of neoprene elastomer.

#### **10.3.4.13 Ethylene propylene elastomer**

No advantage can be gained by postcure heat treatment of ethylene propylene elastomer.

#### **10.3.4.14 Polyolefin**

No advantages in properties or life cycle result from heat treating or a postcure operation for polyolefin; however, loss of properties can occur.

#### **10.3.4.15 Silicone elastomers**

Postcure at or slightly above extended service temperatures in a circulating air oven will enhance all of the positive aspects and properties of silicone elastomers.

### **10.3.5 Welding**

#### **10.3.5.1 Austenitic stainless steel**

Austenitic stainless steels are readily weldable by most processes. Major weld-design considerations include cleaning of weld surfaces, minimizing residual stress (leads to distortion, cracking, or both) with appropriate choice of fitup and heat input, appropriate choice of filler metal (usually type ER308L for 304L and ER316L for 316L) to provide slightly ferritic weld to avoid hot cracking and inhibit carbide precipitation, and adjusting heat input to minimize precipitation in the heat-affected zone of the weld.

#### **10.3.5.2 Carbon steel**

The low-carbon steels can be easily welded or brazed by a number of conventional electric or gas methods if the impurity levels are kept low. Welding of lower grades or free-machining grades should

be performed only when the finished components will be in the stress-free state. Although no thermal pretreatment of the weld area is normally required, the area should be free of oxide, dirt, and grease. Brazing using fluxes and copper alloy filler materials is easily accomplished with low-carbon steels. An intermediate temperature stress relief operation may be required after welding on complex geometries or if uncertainty exists about the in-service stress state of the component. If postwelding heat-stress relief is planned, the weld area should be thoroughly cleaned before heating.

The medium-carbon steels can also be readily welded and brazed if care is taken in designing and preparing the weld. Typically, the materials to be welded should be similar in composition and should be normalized or annealed before welding. Because these materials are frequently used for their hardenability, care must be taken to avoid quenching the weld area. Hardening in the weld area will be accompanied by a ductility loss which can lead to cracking or other forms of failure. Because any large mass adjacent to the weld can act as a sufficient heat sink to quench the weld, the components should be preheated before welding. Gas or electric arc welding with fluxes can produce good welds. Inert gas shielded arc welding may be used to avoid scale formation, but it is more expensive. Resistance welding is more difficult because of the high local heat and the propensity for cracking in the area adjacent to the weld. Brazing with copper alloys and fluxes is common and can be performed on these materials in either the hardened, tempered, or normalized state. After welding, the heat-affected zone surrounding the weld area may require stress relief or tempering, especially if this area will be stressed when placed in service. If care is used to eliminate all stress risers in the design of a weld, welded components can be hardened and tempered as a unit. However, this task will usually require a localized stress relief around the weld area that can be accomplished with a flame or heat tape or blanket. Again, before any postwelding heat treatment, the entire area should be thoroughly degreased and descaled.

### **10.3.5.3 High-strength steel**

These steels are referred to as heat-treatable low-alloy (HTLA) steels. Because of their susceptibility to hydrogen-assisted cracking, these steels are generally welded in the annealed or overtempered condition, and the entire weldment is then heat treated to the desired strength or hardness level. These steels can be readily joined using the shielded metal arc and gas shielded arc welding processes if a minimum preheat and interpass temperature to prevent cracking is maintained and low-hydrogen procedures are employed. The preheat and interpass temperature is dependent on alloy content, heat-treatment condition, availability of hydrogen, joint thickness, and restraint. Care must be given during postweld heat treatment. In some alloys, the weld must not be cooled to room temperature until after it is given a thermal treatment to avoid cracking. These alloys can be brazed using processes, procedures, and filler metals commonly used for carbon steels. When the steel is to be quenched and tempered, brazing and hardening operations can be combined. This information is based on material from Ref. 18.

### **10.3.5.4 Aluminum alloy 6061**

Good weldability is an important characteristic of Aluminum 6061. This alloy is generally readily weldable by all of the usual welding processes. However, some consideration must be given to the special qualities of the alloy. It readily forms an oxide that must be removed by flux, welding arc in an inert atmosphere, mechanical, or chemical means. Also, aluminum does not undergo any color change at welding temperatures, causing difficulty in judging when the metal is close to its melting point. Finally, its high thermal conductivity and electrical conductivity impose special requirements for fusion and resistance welding, respectively. Inert gas shielded arc is recommended because flux is not necessary. Resistance welding using special equipment is satisfactory. The strength of heat-treated alloys

drops to a level comparable to that of a nonheat-treated alloys in the vicinity of the weld. In all cases, the parts must be reaged. When distortion will allow, solution treating and aging will provide maximum properties.

#### **10.3.5.5 Insulating board (cellulosic fiber)**

Welding technology is not appropriate for cellulosic fiber. Excellent bonding can be achieved.

#### **10.3.5.6 Ceramic fiber materials**

Not applicable.

#### **10.3.5.7 Fir plywood**

Not applicable.

#### **10.3.5.8 Redwood**

Not applicable.

#### **10.3.5.9 Urethane foam**

Urethane foams are not generally joined by heat methods because only inferior bonds can be achieved. Bonding methods work well.

#### **10.3.5.10 Silicone foam**

Bonding methods, rather than heating methods, are used to join these foams.

#### **10.3.5.11 Fluoroelastomer**

Not applicable.

#### **10.3.5.12 Neoprene elastomer**

Neoprenes generally are not joined by heat methods. Unlike thermoplastics, neoprenes do not soften or melt when heat is applied. Generally, failure from heat is caused by hardening and loss of resiliency.

#### **10.3.5.13 Ethylene propylene elastomer**

These materials can be heat joined, but resulting bond strengths are relatively poor. Molding to shape is a superior method.

#### **10.3.5.14 Polyolefin**

Polyolefins, like most thermoplastics, can be joined, although in the case of high-molecular-weight materials the resulting bond strengths may be relatively poor. Conventional hot gas plastic welding and various types of heat sealing can be successfully employed on the lower-density and lower-molecular-weight, high-density materials. The higher-molecular-weight polyolefins are most

successfully joined by butt- or friction-welding techniques. Crosslinked or vulcanized polyolefins cannot be joined by techniques in which the substrate undergoes a melt transition.

#### **10.3.5.15 Silicone elastomer**

Not applicable.

### **10.3.6 Bolting**

#### **10.3.6.1 Austenitic stainless steel**

Galling (sticking together of mated/sliding metal surfaces) is a typical problem encountered in the bolting of Types 304L and 316L stainless steel components with traditional threaded bolt-and-nut components of matching composition. Galling can be mitigated with appropriate lubricants or with material selection. An example of the latter is choosing Type 304L stainless steel bolts with Type 303 steel nuts. Type 303 steel is very similar to Type 304L, except it is intentionally alloyed with small amounts of carbon, phosphorus, and sulfur to make it somewhat brittle on a microscopic scale; therefore, its surface tends to crumble during motion, thereby preventing sticking. In any case, bolting of Types 304L and 316L steel components should be performed with materials as similar as possible in composition to the primary alloy to avoid galvanic corrosion. A discussion of galling of threaded components can be found in Ref. 19.

### **10.3.6.2 Carbon steel**

Many grades of steel bolts are used for fastening plain carbon steel. Depending on their strength levels, bolts should be slowly torqued to avoid impact loading. A lubricant or rust chaser should be used to minimize galling and corrosion. Avoid the use of dissimilar metal bolting material (i.e., stainless steel).

### **10.3.6.3 High-strength steel**

Many of these steels are used to make bolts and other fasteners. Because these materials are susceptible to stress corrosion in the hardened condition, use fasteners of the same (or very similar) alloy. Threaded fasteners fabricated with very high-strength materials ( $S_u > 170$  ksi) may be particularly susceptible to stress corrosion cracking, especially when subjected to high preloading. High strength alloys which cannot be completely through-hardened (such as AISI 4140) should not be used for large diameter fasteners.

### **10.3.6.4 Aluminum alloy 6061**

Mechanical joining of aluminum by fasteners poses no real technical problems. Generally, rivets are selected from a material to closely match the properties of the material to be joined. For bolts or rivets, some consideration must be given to coating the fastener to prevent galvanic corrosion of the part. Some form of plating, such as nickel, zinc, or cadmium, is used, depending on the environment.

#### **10.3.6.5 Insulating board (cellulosic fiber)**

Bolting or fastening technology are not effective. Bonding should be used.

#### **10.3.6.6 Ceramic fiber material**

The brittle nature of these materials prohibit effective use of fasteners.

#### **10.3.6.7 Fir plywood**

Plywood can be fastened to itself by nails, screws, and bolts. When using bolts, care must be exercised in making the bolt holes. If the hole is too large, the stress will be distributed nonuniformly. If the hole is too small, inserting the bolt can split the wood. With the other fasteners, the holding strength may degrade with time, rapidly if the wood is subjected to periods of alternating high and low humidity.

#### **10.3.6.8 Redwood**

Redwood can be fastened with nails, screws, spikes, staples, or bolts. When using bolts, care must be exercised in making the bolt holes. If the hole is too large, the stress will be distributed nonuniformly, and inserting the bolt in to a hole that is too small can cause the wood to split. Holding strength may degrade with time when using other fasteners, rapidly if the wood is subjected to periods of alternating high and low humidity.

#### **10.3.6.9 Urethane foam**

Adhesive bonding should be used.

#### **10.3.6.10 Silicone foam**

Compression fit or adhesive bonding should be used.

#### **10.3.6.11 Fluoroelastomer**

Compression fit or adhesive bonding should be used.

#### **10.3.6.12 Neoprene elastomer**

Compression fit or adhesive bonding should be used.

#### **10.3.6.13 Ethylene propylene elastomer**

Compression fit or adhesive bonding should be used.

#### **10.3.6.14 Polyolefin**

Compression fit, welding, or adhesive bonding should be used.

### **10.3.6.15 Silicone elastomers**

Compression fit or adhesive bonding should be used.

## **10.3.7 Bonding**

### **10.3.7.1 Austenitic stainless steel**

For situations where stainless steel is required, it is doubtful that bonding methods other than fusion welding, brazing, or soldering are appropriate. Some materials, such as epoxies, may provide some moderate bond strength if surface preparation (oxide removal or anchor profile) of the alloy is adequate.

### **10.3.7.2 Carbon steels**

Good bonds can be achieved with good cleaning and surface preparation of the steel surface; however, bonding of steel components with glue or similar adhesives is not a common practice for containers because of the low upper limit on bond strength achievable with this approach.

### **10.3.7.3 High-strength steel**

High-strength steels can be bonded quite readily. As with other metals, surface preparation, the type of adhesive used, and the cure cycle determines the strength of the bond. Preparing a surface by methods such as grit blasting offers mechanical interlocking, which generally increases bond strength. Degreasing before grit blasting is recommended to prevent both contamination of the grit and driving contaminants farther into the surface. Blowing off the grit and degreasing the surface after grit blasting

is also recommended to remove any grit or oils deposited on the surface. Degreasing can be accomplished by solvent wiping for large surfaces where immersion is not feasible or by ultrasonic aqueous cleaning where immersion is possible. This type of treatment gives good bond strengths. Acid etching or anodization techniques can also be employed to give superior bond strengths; however, waste disposal concerns must be addressed if this type of treatment is used.

Selecting an appropriate adhesive is an issue that one must address. One must determine whether flexibility, tensile strength, compressive strength, and toughness are needed and then select the appropriate adhesive. Cure cycle is also an important consideration; heat curing may be necessary to obtain full cure and better properties. However, in some situations heat curing may not be feasible because of inconvenience, the large mass making it difficult to heat evenly, or differences in thermal expansion coefficients of materials being bonded. Room-temperature cures can be used in these situations but may require a tradeoff in properties.

#### **10.3.7.4 Aluminum alloy 6061**

Bonding of aluminum parts is quite common. Depending on the strength requirements of the bond, surface preparation can be quite simple. For low-strength bonds, the only preparation necessary might be roughening the surface with a wire brush. For higher-strength bonds, more rigorous preparations are required. The surface must be degreased and, generally, the oxide layer must be removed by etching. Sulphuric, phosphoric, oxalic, or chromic acid anodizing are recommended before bonding. Chromate preparation is falling into disfavor because of the hazardous chromate waste, and the acid-anodizing method, although effective, is difficult to run and monitor. However, bond strengths and bond life can be quite acceptable, especially in a mild- to low-humidity environment.

#### **10.3.7.5 Insulating board (cellulosic fiber)**

Insulating board components can be joined as desired by adhesive bonding to form the required assembly configuration. Most common construction adhesives curing at room temperature will produce a bond line of adequate strength to force failure of the insulating board. Epoxy adhesives may be used when service temperatures for the bond line approach the decomposition temperature of the board itself.

#### **10.3.7.6 Ceramic fiber material**

Should bonding of ceramic fiber materials be required, a number of adhesives, and cements, both organic and inorganic are available. The manufacturer or supplier should be consulted for a particular application.

#### **10.3.7.7 Fir plywood**

Adhesive bonding can be used to supplement assembly of plywood to either itself or heavier wood members with nails or other fasteners to provide better stress distribution over the joint area. Through proper selection and use of an adhesive, an efficient and rigid adhesive-bonded joint can be as strong as or stronger than the wood itself, so some applications may not require additional fastening. Plywood should be selected with few unrepaired defects and with a rough sanded face. Use of surfacing techniques that damage the wood structure can lead to premature bondline failure. One should probably purchase plywood with the appropriate texture for bonding. Glued joints should not be designed primarily to transmit load in tension normal to the plane of the plywood sheet because of the low radial and tangential tensile strength of wood. Glued joints should be arranged to transmit loads through shear planes that are parallel to each other. Sufficient area must be provided between the plywood and the flange member of

box beams and the plywood and the stringers of stressed skin construction to avoid shear failure perpendicular to the grain in the face veneer, in the adjacent ply, or in the wood member itself because shear strength across the grain is less than it is parallel to the grain. Enough pressure should be uniformly applied to the bond joint to distribute the adhesive evenly and to keep the adherents in intimate contact. Do not damage the wood while applying pressure. Best service in wood joints is achieved if the wood is bonded at a moisture content as close to that anticipated in the joint in service as is possible to minimize the tendency for the joint to separate because of dimensional changes.

Phenol formaldehyde and urea formaldehyde adhesives are two-part thermosetting systems and are typically heat cured. They can be formulated for room-temperature curing; however, room-temperature-curing phenolics are acid catalyzed and are not widely used because of potential damage to the wood. Room-temperature-cured urea adhesives have limited water and heat resistance and should be used only for interior applications. Resorcinol resins are also two-part systems that provide durable room-temperature-cured joints suitable for exterior and severe exposures. Two-part epoxy leads to a strong bondline but does not provide adequate moisture resistance. Epoxy can be used as a gap-filling adhesive because of the low shrinkage and outgassing during cure. Aqueous emulsions of polyvinyl acetate are convenient, one-part, high-strength adhesives. They have limited resistance to moisture and can creep in joints under stress. Mastic adhesives can be used for thick and variable-thickness gluelines. They are usually solvent-based elastomers and do not provide much strength.

#### **10.3.7.8 Redwood**

Redwood generally bonds very easily using a wide variety of glues. The moisture content of the wood has much to do with the final strength of the bond and the dimensional stability of the glued members. However, redwood has a high dimensional stability, somewhat mitigating that consideration.

Also, if the wood has a moisture content of less than 12%, a satisfactory bond can generally be achieved. A wide range of products-animal, vegetable, natural and synthetic rubber products, and epoxies-are available to glue wood to wood. Good results can also be obtained by using some products to fill in chips or cracks in the wood.

#### **10.3.7.9 Urethane foam**

Foam structures are not generally bonded because of their highly porous surface. Compression fit is superior when possible.

#### **10.3.7.10 Silicone foam**

Foam structures are not generally bonded because of their highly porous surface. Compression fit is superior when possible.

#### **10.3.7.11 Fluoroelastomer**

Sanding may be needed as part of surface preparing fluoroelastomer for adhesive bonding. Bonds to metal substrates are best made during vulcanization using appropriate primers; postvulcanization bonds also require primers, although strengths are generally lower.

#### **10.3.7.12 Neoprene elastomer**

Neoprene can be bonded using two different methods. One method is to cure or vulcanize the uncured elastomer on a metallic or fabric surface. This method is preferred if higher bond strengths are

required. A large variety of primers or bonding agents are available on the market to enhance bond strengths obtained when bonding elastomers to metal surfaces. These are generally applied to a clean metal surface. The uncured rubber is then laid on the surface and cured in place. Contact between the uncured rubber and the metallic surface must be made to ensure a good bond. When strength requirements are not as stringent, neoprenes may also be joined by using a neoprene adhesive, which is a neoprene compound dissolved in solvent. This method is generally used when bonding neoprene to itself.

#### **10.3.7.13 Ethylene propylene elastomer**

Bonding ethylene propylene elastomer with an adhesive can be performed, but the surface must be prepared with aggressive reagents and the resulting bonds are usually of low strength.

#### **10.3.7.14 Polyolefin**

Bonding polyolefin with an adhesive can be performed, but the surfaces must be prepared with aggressive reagents and the resulting bonds are usually of low strength.

#### **10.3.7.15 Silicone elastomer**

Bonding silicone elastomer with an adhesive can be performed, but the surfaces must be prepared with aggressive reagents and the resulting bonds are usually of low strength.

## **10.3.8 Plating**

### **10.3.8.1 Austenitic stainless steel**

Austenitic stainless steels can be plated to generate specific surface properties, but care must be exercised in achieving an intimate bond between stainless steel and the plating material. An oxide film forms on these alloys in many environments, including spontaneous formation in air, and this film interferes with bonding for many plating situations. Some aqueous baths can provide a surface appropriate for plating. Vacuum furnaces with hydrogen for reducing oxides followed by sputtering or vapor deposition is also a common method of plating stainless steel.

### **10.3.8.2 Carbon steel**

Permanent protection of the low- and medium-carbon steels is neither cheap nor easy, but it can be accomplished if adequate care is taken in preparing the surfaces and if quality materials are used in the plating process. For a more or less decorative corrosion preventative, chromium plating is sometimes used, generally with a good undercoating of 0.05 mm of copper and 0.025 mm of nickel. Cadmium, tin, and zinc can all be applied. Metal-sprayed coatings such as zinc and aluminum are sometimes used for protection. These coatings require dry blasting of the surface before coating and sealing by wire brush and chemical treatment immediately after spraying. Probably the most common form of protection for these materials is galvanizing, which involves dipping a chemically cleaned steel part into a bath of molten zinc under controlled time and temperature conditions that cause the zinc and the steel to react, forming a very adherent layer of zinc-iron compounds with free zinc at the surface. The layer of free zinc provides some barrier protection for the substrate and sacrificial corrosion protection when adequate moisture is available. Precautions for galvanizing are mainly related to potential for dimensional

instability during rapid temperature changes. Galvanizing (or other coating and plating) should be performed after all forming and welding operations as the coatings can be damaged by deformation or heat and the coated components embrittled by welding. Stress relieving after plating of carbon steels may be required to prevent hydrogen embrittlement.

#### **10.3.8.3 High-strength steel**

Decorative chromium plating is not often required on these steels. When necessary, at least 0.05 mm of nickel or copper and nickel undercoat should be deposited below the thin coat of chromium. Cadmium and zinc plating 0.025 mm thick gives excellent corrosion protection under all normal conditions. The majority of these steels will be above 1000 MPa (145 ksi) tensile strength and require stress relieving at 200°C after plating. If considerable machining stress exists, stress relief before plating is recommended. This task is required to eliminate hydrogen embrittlement, which can cause brittle failure below the calculated tensile strength. This defect becomes more serious as the hardness increases. Although hydrogen embrittlement can be entirely removed by the 200°C stress relief treatment, visual proof that this is always carried out on every part is impossible. Because brittle failure of high-tensile-steel parts can be disastrous, it is recommended that parts from 175 to 200 MPa tensile strength be purchased and controlled.

#### **10.3.8.4 Aluminum alloy 6061**

Aluminum 6061 can be plated if specific surface requirements demand it. The oxide layer on the aluminum must be removed to achieve good conduct between the plating material and the aluminum.

**10.3.8.5 Insulating board (cellulosic fiber)**

Not applicable.

**10.3.8.6 Ceramic fiber material**

Not applicable.

**10.3.8.7 Fir plywood**

Not applicable.

**10.3.8.8 Redwood**

Not applicable.

**10.3.8.9 Urethane foam**

Not applicable.

**10.3.8.10 Silicone foam**

Not applicable.

#### **10.3.8.11 Fluoroelastomer**

Because of the flexible nature of elastomers, plating of these compounds is not normally conducted. The elastomer will flex easily, causing cracking of the plating.

#### **10.3.8.12 Neoprene elastomer**

Because of the flexible nature of elastomers, plating of these compounds is not normally conducted. The elastomer will flex easily, causing cracking of the plating.

#### **10.3.8.13 Ethylene propylene elastomer**

Because of the flexible nature of elastomers, plating of these compounds is not normally conducted. The elastomer will flex easily, causing cracking of the plating.

#### **10.3.8.14 Polyolefin**

Surface decorating operations are something of a challenge with polyolefins because of their extremely low surface energies, especially the highly crystalline polyethylenes. Various inks and paints are available that can wet and mark polyolefin surfaces, but all of these methods should be regarded as temporary, because light abrasion, such as that resulting from normal handling, invariably degrades the quality of the markings. Coatings, such as paints that are intended to be permanent, require a surface pretreatment. Many techniques have been devised for such surface treatment, the simplest being surface roughening, which is of limited use and worth. Two very widely used surface preparation methods are flame treatment and corona discharge, both of which leave an active surface capable of accepting a

permanent coating. Chemical etching, which has also been used with success, has fallen out of favor because of concerns about waste disposal. Because polyolefin systems are usually of relatively low stiffness, rigid surface coatings may be subject to cracking as the substrate materials are mechanically or chemically stressed.

#### **10.3.8.15 Silicone elastomer**

Because of the flexible nature of elastomers, plating of these compounds is not normally conducted. The elastomer will flex easily, causing cracking of the plating.

### **10.3.9 Painting**

#### **10.3.9.1 Austenitic stainless steel**

Austenitic stainless steels may be painted (for cosmetics or further corrosion protection), but adhesion is often poor unless the surface has been roughened to provide an anchor profile and unless the type of paint or coating is selected with adhesion to a passive film on stainless steel in mind.

#### **10.3.9.2 Carbon steel**

Painting plain carbon steel is a common method used to improve the cosmetic appearance and increase resistance to atmospheric corrosion. Any improvement in atmospheric corrosion resistance depends critically on the quality of the paint or coating chosen (durability, permeation resistance, and ultraviolet light resistance) and the degree of surface preparation imparted to the steel. Before application of a paint or coating, the steel must be dry and free of any scale, grease and oil, and other debris. This

task can be accomplished by chemical cleaning, grit blasting the surface, or both. In addition, the steel must have an appropriate anchor profile (roughness) to hold the paint or coating. This goal can be accomplished chemically, but mechanical methods such as grit blasting are usually superior. The choice of paint or coating depends on the expected environment and desired service life. Because of the labor-intensive operations necessary for surface preparation, painting or coating the carbon steel may add significantly to the cost even above that of the paint or coating.

### **10.3.9.3 High-strength steel**

When properly controlled, grit blasting is the best method for cleaning and preparing the surface of high-strength steel as it leaves a roughened surface to act as a key for the subsequent film. The surface must be brushed to remove the dust caused by blasting, and the subsequent treatment must be carried out as soon as possible after blasting because the freshly prepared surface can corrode very rapidly.

Phosphate treatment acts as an excellent paint base and is in itself a corrosion-resistant film and an oil-retentive surface. In this condition and if the oil film is maintained, long corrosion-free life can be achieved under indoor conditions. Steels above 1000 N/mm<sup>2</sup> tensile strength should be stress relieved to eliminate hydrogen embrittlement.

The phosphated surface must be oven dried before application of zinc silicate or chromate primer. For best results, top coats of paint should be applied as quickly as possible after the primer.

#### **10.3.9.4 Aluminum alloy 6061**

Aluminum 6061 can be painted to enhance appearance or for further corrosion protection if desired. Many of the considerations of bonding apply to painting aluminum surfaces. For high-finish surfaces, roughening with a metal brush or chemical etching is generally required to improve paint adhesion. Best paint performance can be obtained with epoxy-polyamide paints on an anodized surface.

#### **10.3.9.5 Insulating board (cellulosic fiber)**

Insulating board can be painted to reduce dust or handling damage and for identification.

#### **10.3.9.6 Ceramic fiber material**

Ceramic fiber materials can be painted for identification.

#### **10.3.9.7 Fir plywood**

Plywood intended for continuous outside exposure should be protected from ultraviolet degradation. Plywood can be finished with water-repellent preservatives, stains, or house paint. Water-repellent preservatives are formulated to penetrate the wood surface and are used if the natural finish is to be retained. Shellac or varnish will tend to crack or peel because they do not penetrate the wood. Acrylic latex, oil-based, or solvent-based stains provide adequate weathering protection. Acrylic latex paint is recommended and should be used with a primer and a top coat. Fir plywood must be sealed with either shellac or a special fir-plywood primer before painting because of the wild grain. Do not use oil or alkyd paints because they tend to flake. Prefinished plywood and plywood treated for protection

against fire or decay can be purchased. Plywood is usually treated after the plies are glued so that only panels made with exterior glue are treated. Wood preservation techniques for plywood are the same as for ordinary lumber.

#### **10.3.9.8 Redwood**

Paints or other finishes, such as varnishes and water repellents, can be applied readily to redwood. Such finishes can be used for cosmetic reasons or to protect the wood. Moisture content of the wood should be low, less than 12%. Applying paint or another finish to redwood is usually straightforward. Consideration should be given to the environment in which the wood will be placed. If the environment is wet or at an extremely high level of humidity, a water repellent should be used as a first step, then a primer and the final finish can be applied. Best results are obtained by using two to three coats of varnish, lacquer, or paint. However, because redwood has a high natural resistance to decay, good results can be obtained with fewer coats.

#### **10.3.9.9 Urethane foam**

Not applicable.

#### **10.3.9.10 Silicone foam**

Not applicable.

#### **10.3.9.11 Fluoroelastomer**

As with plating, the flexible nature of elastomers does not promote good coating characteristics. The elastomer flexes easily, causing cracking of the coating.

#### **10.3.9.12 Neoprene elastomer**

As with plating, the flexible nature of elastomers does not promote good coating characteristics. Neoprene flexes easily, causing cracking of the coating.

#### **10.3.9.13 Ethylene propylene elastomer**

As with plating, the flexible nature of elastomers does not promote good coating characteristics. The elastomer flexes easily, causing cracking of the coating.

#### **10.3.9.14 Polyolefin**

Painting of polyolefin is recommended because of poor adhesion.

#### **10.3.9.15 Silicone elastomer**

As with plating, the flexible nature of elastomers does not promote good coating characteristics. The elastomer flexes easily, causing cracking of the coating.

## 10.4 QUALITY

### 10.4.1 Austenitic Stainless Steel

The composition and properties of austenitic stainless steel are controlled by a number of ASTM specifications. Perhaps the most significant composition feature is the carbon content, which separates the "L"-grade material (0.030 or 0.035% carbon maximum, depending on allowances for product form and analysis details) from the standard grade (up to 0.08% carbon allowed). Other elements are controlled within specific ranges or with maximums, and experience shows that meeting all of the composition requirements for these materials is both technically significant and relatively easy for vendors. Deviations from the composition requirements should not be lightly regarded.

Some specifications also include requirements for mechanical properties (usually minimum strength or ductility, maximum hardness), heat treatment (usually solution treatment or annealing), or other requirements for fabrication. In addition, each specification usually contains a list of other applicable specifications that are useful to or invoked by the one at hand. It is beyond the scope of this document to list every applicable specification (ASTM or other organizations); however, depending on product form and the other requirements desired by the customer, several common ASTM specifications for these alloys include the following:

- *ASTM A666: Standard Specification for Austenitic Stainless Steel Sheet, Strip, Plate, and Bar*
- *ASTM A484: General Requirements for Stainless/Heat-Resisting Bars, Billets, and Forgings*

- *ASTM A240: Standard Specification for Heat Resisting Chromium and Chromium-Nickel Stainless Steel Plate, Sheet, and Strip for Pressure Vessels*
- *ASTM A743: Standard Specification for Castings, Iron-Chromium, Iron-Chromium-Nickel, Corrosion Resistant, for General Application*
- *ASTM A744: Standard Specification for Castings, Iron-Chromium-Nickel, for Severe Service*
- *ASTM A276: Standard Specification for Stainless and Heat-Resisting Bars and Shapes*

A common corrosion test specification used for austenitic stainless steels is ASTM A262 (*Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels*). This specification contains a number of recommended practices for conducting standardized corrosion tests. Experience shows that requirement of passing Practice A (oxalic acid etch) and Practice C (boiling nitric acid test, limit 0.020 in. per month average in at least three test periods) for each heat or lot results in quality material with maximum corrosion performance characteristics. This test is most common for "L"-grade materials in service where corrosion resistance is typically the most important, and it is a restrictive requirement in that many vendors do not have immediate access to this data for the materials on hand. Therefore, testing at an independent laboratory is required, which increases costs and adds time to the procurement schedule. However, the requirements of the corrosion tests are not overly demanding, and quality material should pass these tests without deviation. For very mild service conditions, these tests are sometimes waived or minimized (e.g., requiring only Practice A). The necessity of this requirement (or others imposed by the various ASTM specifications) must be judged for each application, and it is recommended that input from an individual experienced in these tests be obtained for each procurement.

## 10.4.2 Carbon Steel

The quality of the carbon steels can be roughly assessed from the phosphorus and sulphur contents, which should be as low as possible unless specifically added to aid machinability. Any other residual elements present could have an adverse effect on welding because they tend to increase hardenability. The AISI/SAE designation and standard practices used to describe plain carbon steel are not specifications and contain only a portion of the information (namely, range of chemical composition) needed to properly describe a plain carbon steel product for procurement. ASTM publishes the most widely used standard specifications for procuring plain carbon steels; they are fairly complete and are generally oriented toward performance of the fabricated end product. Most ASTM specifications for bars, wires, billets, and forgings and some specifications for sheet products incorporate the AISI/SAE designations indicating chemical composition. However, the specifications for plate and structural shapes usually specify the limits and ranges of chemical composition directly without using the AISI/SAE designation. Some of the commonly used ASTM specifications for procuring carbon steels are as follows:

- A108: Standard-quality, cold-finished carbon steel bars
- A576: Special-quality, hot-rolled carbon steel bars
- A659: Commercial-quality, hot-rolled carbon steel sheet and strip
- A611: A, B, C, or E Grade Cold Rolled Structural Quality (0.2% carbon, 0.6% manganese)

The Aerospace Materials Specifications (AMS) published by SAE are also complete specifications that can be used for procurement; however, because these specifications pertain to materials used in the aerospace industry, they may contain unnecessarily strict requirements on such things as mechanical properties that may require special processing methods that will cost more than steels of similar

composition intended for other applications. An exhaustive list of ASTM, AMS, and other specifications are given in Ref. 5.

### 10.4.3 High-Strength Steel

A variety of forms, grades, and classes of high-strength, low-alloy, medium-carbon steels are available commercially. Many of these materials are used in the aerospace industry. Hence, many specifications have been developed by the government (Military Specifications) or by other groups, such as the Aerospace Materials Specifications published by the SAE, Inc., that are suitable for use in procuring these materials. Some of these are written for specific uses; others refer to specific forms. Still others are more general in nature and may not be sufficient for procurement without other information. Some specifications call out the alloy manufacturing process, which may have an indirect effect on the physical or chemical properties of the materials. Because AISI 4340 is considered a standard for medium-carbon, low-alloy, high-strength steels, sample specifications appropriate for procuring this material are as follows:

- AMS 6359: *Steel, Sheet, Strip, and Plate (0.8 Cr -1.8Ni - 0.2SMo 0.38-0.43C) (SAE 4340)*
- Mil-S-8 844: *Steel Bars, Reforging Stock, and Mechanical Tubing, Low Alloy, Premium Quality*
- Mil-S-5000: *Steel Chrome-Nickel-Molybdenum (E4340) Bars and Reforging Stock*

Most specifications reference other standards or specifications for measuring properties, determining composition, detecting flaws, etc.

#### **10.4.4 Aluminum Alloy 6061**

The systems for designating alloys and tempers are covered by national standard ANSI H35.1-1982. The rules by which alloy distinctions are also made and designations assigned are a part of this document. Aluminum association composition limits for wrought aluminum alloys provide a chemical purity standard. Because the principal use of this alloy has been heavy-duty structures requiring good corrosion resistance (e.g., aircraft, railroad cars, pipelines, marine craft, etc.) this alloy is sold to consistently high standards. Specifications from the aerospace industry will provide for maximum control.

#### **10.4.5 Insulating Board (Cellulosic Fiber)**

A variety of standard sizes and grades of insulating board are available from manufacturers of wood-based products. Examples of board grades available include roof insulating, sheathing, building, shinglebacker, and sound deadening. Sizes (width and length) vary from 1 ft by 1 ft to 4 ft by 12 ft, and thicknesses vary from 1/2 to 3 in. when manufactured in accordance with ASTM C208, specification for commercial products. More restrictive specifications do not exist.

Standard specifications and methods of testing for insulating board products include the following:

- *ASTM C208: Standard Specification for Insulating Board (Cellulosic Fiber), Structural and Decorative*
- *ASTM D1554: Definition of Terms Relating to Wood-Base Fiber and Particle Panel Materials*

- *ASTM D2277: Specification for Fiberboard Nail-Base Sheathing*
- *ASTM C209: Standard Method of Testing for Insulating Board (Cellulosic Fiber), Structural and Decorative*
- *ASTM C165: Measuring Compressive Properties of Thermal Insulations*

#### **10.4.6 Ceramic Fiber Material**

The ceramic fiber materials industry has not matured to a level providing uniform commercial specifications. However, the rigorous conditions under which these materials are normally used have required that the vendors provide reliable physical and mechanical property data. The references listed for this material, ASTM standards, and vendor literature provide the best basis for specification preparation.

#### **10.4.7 Fir Plywood**

Several ASTM standards concern the measurement of flexural (D3034), tensile (D3500), compressive (D3501), shear strength (D2718), shear modulus (D3044), and toughness (D3499). ASTM D1038 lists standard definitions used in relation to plywood. The quality of the plywood will depend on the adhesive used and the source of the wood. The APA-adopted PS1 standard governs the production and grading of plywood. The standard includes specifications concerning the requirements for species, grade, thickness of veneers and panels, glue bonds, moisture content, standard definitions of terminology, suitability, and performance test specifications. The stamp on plywood indicates the standard; not all APA plywood conforms to the PS1 standard. APA performance-rated grades and a Federal Housing

Association standard exist. Not all imported plywood follows the PS1 standard, and those that do will not carry the APA stamp. The stamp also lists a mill number that indicates the manufacturer of the plywood; this number can be useful if an exact match to existing stock is desired.

#### **10.4.8 Redwood**

Redwood stock from the heartwood is moderately light in weight while possessing moderate strength stiffness and hardness. The heartwood has high decay resistance, is easily worked, is straight grained, and shrinks and swells comparatively little. In addition to normal lumber specifications, the purchaser should avoid pieces containing the sapwood, which is almost white in contrast to the light cherry to dark mahogany heartwood. The wood splits readily, and structural pieces should be selected with an eye toward avoiding stress points or flaws that can initiate splits.

#### **10.4.9 Urethane Foam**

Most urethane foams produced on the continuous production lines are uniform and easily sampled randomly for properties. Vendor certification of properties measured for their high-volume customers are readily available. Prepackaged "foam in place" kits are more difficult to use in obtaining uniform properties. Vendor instructions must be followed exactly, temperature and humidity limits observed, component shelf life honored, and extreme cleanliness maintained to achieve uniform properties. Most kits are not intended for applications where predictable compression set and strength or high-temperature stability are essential.

#### **10.4.10 Silicone Foam**

Most silicone foams produced on proprietary production lines are amazingly uniform and easily sampled randomly for properties. Vendor certification of properties measured for their high-volume customers are readily available. Prepackaged "foam in place" kits are more difficult to use in obtaining uniform properties. Vendor instructions must be followed exactly, temperature and humidity limits observed, component shelf life honored, and extreme cleanliness maintained to achieve uniform properties. Most kits are not intended for applications where predictable compression set and strength or high-temperature stability are essential.

#### **10.4.11 Fluoroelastomer**

Physical properties of elastomers—whether determined for quality control, for compliance with specifications, or for research and development—are a challenge. Tests used for other solid materials are seldom applicable to elastomers because of the time and temperature dependence of their properties. Ultrahigh deformations and low moduli of elastomers require that test equipment be accurate at higher displacements and lower forces than are needed for most engineering materials. Good reproducibility can be obtained only from thoroughly developed methods, which is one of the reasons for the many standardized tests used for elastomers.

A variety of standardized tests are required to characterize the fluoroelastomers. Raw gums are tested for Mooney viscosity (ASTM D 1646) and specific gravity (ASTM D 297). When compounded, the stocks are tested for Mooney cure and scorch (ASTM D 1646) and oscillating-disk rheometer cure rate (ASTM D 2705). The vulcanizates are evaluated as to the as-cured physical properties (ASTM D 412 and D 1414), aged physical properties (ASTM D 573), compression set (ASTM D 395), and fluid

aging (ASTM D 471). Low-temperature properties tested are low-temperature retraction TR10 (ASTM D 1329), torsional or dynamic modulus (ASTM D 1053), and brittle point (ASTM D 746). Occasionally, special tests may be required, depending on the application. Some examples are tear resistance (ASTM D 624), electrical strength (ASTM D 149), dielectric constant and dissipation factor (ASTM D 150), and volume resistivity (ASTM D 257). Reproducibility of results, especially between laboratories, is very poor for elastomers unless the standards are carefully followed. Equal attention to detail is required in interpretation of test results.

Many good resources for information on fluoroelastomers are available, including Refs. 12-14. Product literature from the vendors is especially useful.

The O-ring manufacturer needs to know the inside diameter, the cross-sectional diameter, the shape, and the compound (formulation) from which it is to be made. The manufacturer must provide batch identification on the product that will allow complete traceability at virtually any time during its life cycle. The manufacturer's internal Quality Assurance system should allow for the retrieval of data on the physical properties of any given lot of product from compounding to finished product. Actual lot values for hardness, tensile strength, ultimate elongation, and specific gravity are a minimum.

#### **10.4.12 Neoprene Elastomer**

The O-ring manufacturer needs to know the inside diameter, the cross-sectional diameter, the shape, and the compound (formulation) from which it is to be made. The manufacturer must provide batch identification on the product that will allow complete traceability at virtually any time during its life cycle. The manufacturer's internal Quality Assurance system should allow for the retrieval of data

on the physical properties of any given lot of product from compounding to finished product. Actual lot values for hardness, tensile strength, ultimate elongation, and specific gravity are a minimum.

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A variety of standardized tests are required to characterize the neoprene elastomers. Raw gums are tested for Mooney viscosity (ASTM D 1646) and specific gravity (ASTM D 297). When compounded, the stocks are tested for Mooney cure and scorch (ASTM D 1646) and oscillating-disk rheometer cure rate (ASTM D 2705). The vulcanizates are evaluated as to the as-cured physical properties (ASTM D 412 and D 1414), aged physical properties (ASTM D 573), compression set (ASTM D 395), and fluid aging (ASTM D 471). Low-temperature properties tested are low-temperature retraction TR10 (ASTM D 1329), torsional or dynamic modulus (ASTM D 1053), and brittle point (ASTM D 746). Occasionally, special tests may be required, depending on the application. Some examples are tear resistance (ASTM D 624), electrical strength (ASTM D 149), dielectric constant and dissipation factor (ASTM D 150), and volume resistivity (ASTM D 257).

### **10.4.13 Ethylene Propylene Elastomer**

The O-ring manufacturer needs to know the inside diameter, the cross-sectional diameter, the shape, and the compound (formulation) from which it is to be made. The manufacturer must provide batch identification on the product that will allow complete traceability at virtually any time during its life cycle. The manufacturer's internal Quality Assurance system should allow for the retrieval of data on the physical properties of any given lot of product from compounding to finished product. Actual lot values of hardness, tensile strength, ultimate elongation, and specific gravity are a minimum.

Physical properties of elastomers—whether determined for quality control, for compliance with specifications, or for research and development—are a challenge. Tests used for other solid materials are seldom applicable to elastomers because of the time and temperature dependence of their properties. Ultrahigh deformations and low moduli of elastomers require test equipment to be accurate at higher displacements and lower forces than are needed for most engineering materials. Good reproducibility can be obtained only from thoroughly developed methods, which is one of the reasons for the many standardized tests used for elastomers. Reproducibility of results, especially between laboratories, is very poor for elastomers unless the standards are carefully followed. Equal attention to detail is required in interpretation of test results.

The elastomers are evaluated as to the as-cured physical properties (ASTM D 412 and D 1414), aged physical properties (ASTM D 573), compression set (ASTM D 395), and fluid aging (ASTM D 471). Low-temperature properties tested are low-temperature retraction TR10 (ASTM D 1329), torsional or dynamic modulus (ASTM D 1053), and brittle point (ASTM D 746). Occasionally, special tests may be required, depending on the application. Some examples are tear resistance (ASTM D 624), electrical

strength (ASTM D 149), dielectric constant and dissipation factor (ASTM D 150), and volume resistivity (ASTM D 257).

#### **10.4.14 Polyolefin**

The ASTM Committee D20 on Plastics has published ASTM Standard D4101 for polypropylene and a broad spectrum of standard test methods for physical and mechanical properties of plastics. The Naval Air Systems Command is the Department of Defense (DOD) military coordinating agency for the adoption of AMS for plastics. Approximately 50 SAW AMS 3000 Series documents for plastics such as those on molding compounds, sheet tubing, and foams have been officially adopted for DOD use and should prove useful. For flammability testing, the Underwriters' Laboratories Standards UL94 and UL746 should prove useful.

#### **10.4.15 Silicone Elastomer**

O-rings made with silicone heat-cured rubbers offer a number of unique properties and a much wider range of properties than other elastomer compounds. The purchaser is very dependent upon the integrity and quality assurance of the vendor. As with the other elastomeric materials, the O-ring manufacturer needs to know the inside diameter, the cross-sectional diameter, shape and the compound (formulation) from which it is to be made. The manufacturer must provide batch identification on the product that will allow complete traceability at virtually any time during its life cycle. The manufacturer's internal quality assurance system should allow for the retrieval of data on the physical properties of any given lot of product from compounding to finished product. Actual lot values for hardness, tensile strength, ultimate elongation, and specific gravity are a minimum.

Physical properties of elastomers—whether determined for quality control, for compliance with specifications, or for research and development—are a challenge. Tests used for other solid materials are seldom applicable to elastomers because of the time and temperature dependence of their properties. Ultrahigh deformations and low moduli of elastomers require test equipment to be accurate at higher displacements and lower forces than are needed for most engineering materials. Good reproducibility can be obtained only from thoroughly developed methods, which is one of the reasons for the many standardized tests used for elastomers. Reproducibility of results, especially between laboratories, is very poor for elastomers unless the standards are carefully followed. Equal attention to detail is required in interpretation of test results.

The elastomers are evaluated as to the as-cured physical properties (ASTM D 412 and D 1414), aged physical properties (ASTM D 573), compression set (ASTM D 395), and fluid aging (ASTM D 471). Low-temperature properties tested are low-temperature retraction TR10 (ASTM D 1329), torsional or dynamic modulus (ASTM D 1053), and brittle point (ASTM D 746). Occasionally, special tests may be required depending on the application. Some examples are tear resistance (ASTM D 624), electrical strength (ASTM D 149), dielectric constant and dissipation factor (ASTM D 150), and volume resistivity (ASTM D 257).

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