Subsequent investigations have shown that this law is obeyed for many pure f.c.c. metals, both single- and polycrystalline, and that it applies for changes in strain rate as well as temperature. The Cottrell–Stokes Law is not applicable to b.c.c. materials or to f.c.c. alloys that are strain or precipitation hardened; mixed results have been obtained with h.c.p. materials. Since this law is concerned with the thermal part of the flow stress, it properly applies only when at least one of the two temperatures considered is low enough so that the thermal contribution to the flow stress is significant.

7.3.7 Work softening

Work softening is the name given by Stokes and Cottrell (1954) to an effect that involves straining a specimen first at a low temperature and then at a higher temperature. At the higher temperature, their specimens exhibited a yield point, then a decrease in the load required to continue the deformation, and then continuous deformation at loads equal to those for specimens strained only at the higher temperature. The yield-point effect was not due to diffusion of solutes to dislocations and so must have been an effect of the dislocation structure introduced during the low-temperature deformation. A similar effect was observed by Haasen and Kelly (1957): a small yield-point effect was introduced into a metal sample deformed at a relatively low temperature by fully or partially unloading the specimen and then reloading.

7.4 TOUGHNESS, FATIGUE, AND CREEP

Low-temperature toughness and resistance to creep and fatigue depend on the same basic mechanisms of plastic flow and resistance to dislocation motion as tensile properties, but with added complications. The behaviors of these properties are not as well understood on a microscopic level as tensile property behavior. Most studies have been oriented toward evaluation of practical materials for structural use rather than toward fundamental understanding of the basic mechanisms using pure and single-crystal specimens. The treatment of toughness, fatigue, and creep in this section has the same orientation. Fatigue and creep are discussed as they apply to unnotched specimens. Fatigue in notched and cracked specimens and current practical applications of the concept of toughness are discussed in Chapter 8, Fracture Mechanics.

7.4.1 Toughness

Toughness represents the ability of a material to resist fracture in the presence of large stresses in the neighborhood of a notch, crack, or other stress concentrator and is measurable in special fracture toughness tests. Toughness is an important low-temperature property because the decrease in toughness on cooling that occurs in ferritic steels is the most deleterious effect of cryogenic environments on commonly used structural materials. Toughness is controlled by the basic mechanisms of strength and deformation described in § 7.2, but the relationship between the measured property and the fundamental deformation processes is complex, in contrast to that for tensile properties. The complexity is due to the spatially nonuniform stress and strain fields and multiaxial stresses that occur in toughness testing owing to the use of notched and cracked specimens. A quantitative theoretical treatment of toughness would produce, in contemporary terminology, a calculation of the temperature dependence of the plane strain critical stress intensity, K_{Ic}. This quantity describes the resistance of a material under stress to fractures that begin at a stress concentrator. Such a calculation is currently not available.

The Charpy impact test and other toughness measurements. The Charpy impact test has been used widely in the United States as a low-temperature toughness measurement method for ferritic and stainless steels and their welds (*Annual Book of ASTM Standards*, 1976). In this technique, a heavy pendulum fitted with a special impact fixture called a tup is made to swing into and through a notched bar of specimen material. The energy absorbed by the specimen is related directly to the difference between the height to which the pendulum swings without impacting a specimen and the height reached after fracturing a specimen. Measurements of the deformation produced by the test are commonly made on the fractured specimen. Commercially manufactured Charpy impact testing machines capable of delivering a variety of energies at impact are available. Specimen temperature is controlled by cooling the specimen to the desired temperature by immersion in an appropriate cryogen, such as liquid nitrogen, and then rapidly inserting the specimen into the machine and immediately impacting it. Lower temperatures are reached by using a flimsy, disposable container filled with cryogen around the speci-

men. Internal specimen heating from the high strain rates during fracture limits the effectiveness of these techniques.

Charpy impact energy is plotted as a function of temperature, as shown in Fig. 7.16. Significant scatter is commonly observed in the values of energy absorbed in Charpy tests, but the results of many tests are usually represented by a smooth curve like the one in Fig. 7.16. The transition between high and low values of absorbed energy generally occurs over a fairly narrow temperature range in low-strength (less than about 700 MPa or 100 ksi) ferritic steels. The absorbed energy often changes much more gradually in other materials. Over the last decade, Charpy specimen lateral expansion opposite the notch has replaced the impact energy in some codes and standards. Lateral expansion, measured opposite the notch, is believed to represent more consistently the fracture resistance of materials with a wide range of yield strengths. In some United States codes, a lateral expansion of at least 0.38 mm has been required (see Chapter 8). In Fig. 7.16, lateral expansion is plotted against temperature along with the impact energy. Other quantities observed in a Charpy test that may be plotted against temperature are the percent fibrous (shear) fracture visible on the fracture surface and the lateral contraction at the notch root. Neither of these has been accepted by code-writing groups as a standard test of material quality.

The most severe limitation of the Charpy test is that the results are a qualitative indication of toughness and cannot be used directly in structural design calculations. Other limitations are the scatter often observed in the data, its inability to show the toughness transition in materials that fail by low-energy tearing instead of cleavage, and its inapplicability to nonferrous materials and specimens less than 5-mm thick.

Notch tensile testing is another technique for comparative toughness evaluation. In this method, a notch is machined around the circumference of a cylindrical specimen, or at the edges of a plate specimen, which is then loaded in tension. The notch strength or notch tensile strength is the maximum load sustained divided by the remaining cross-sectional area of the specimen at the notch. The ratio of the notch strength to the ultimate tensile strength or to the tensile yield strength (measured on an unnotched specimen) is an index of the specimen toughness. The notch–yield ratio, that is, the ratio of notch strength to yield strength, is recommended as a more meaningful parameter in a proposed test method (*Annual Book of ASTM Standards*, 1976). Like the Charpy impact technique, the notch tensile method is a qualitative measure of toughness.

A variety of notch depths and root radii have been used by various investigators for notch tensile testing, although the proposed test method referenced above specifies fixed notch dimensions. The stress at the notch root is higher than the average stress far from the notch. The stress concentration factor, K_t, gives the ratio of the
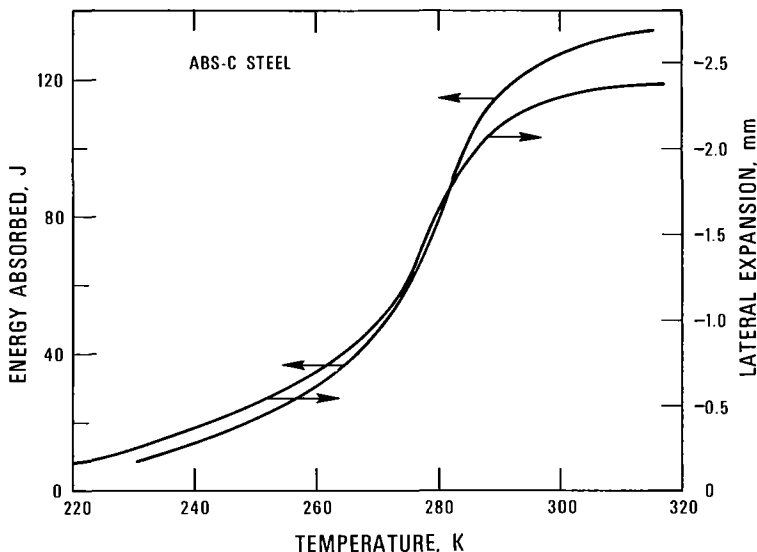

Figure 7.16 Temperature dependence of energy absorbed and lateral expansion in Charpy impact toughness test for A-533-B steel (Gross, 1970).

stress at the notch to the average stress far from the notch. Derivations of stress concentration factors for several notch configurations are given in the classic work by Neuber (1946). A more recent compilation by Peterson (1973) gives stress concentration factor values for a wide variety of notch configurations encountered in structural design.

Ductile-to-brittle transition in ferritic steels. A much-used concept related to toughness is the ductile–brittle transition. Some materials, in practice mainly b.c.c. iron alloys, are capable of ductile behavior at high temperatures but become brittle at low temperatures. The ductile–brittle transition temperature marks this transition. Many structural materials show a gradual transition between brittle and ductile behavior and so do not have a genuine ductile–brittle transition temperature. One theory of the ductile–brittle transition is that the mechanisms of failure by brittle fracture and failure by ductile fracture following yielding are potentially operative at all temperatures (Knott, 1973). These two failure modes are viewed as alternate possibilities; the one that requires lower stress is realized. At high temperatures, the ductile–failure stress is lower; at low temperatures, the brittle–failure stress is lower. The ductile–brittle transition temperature, T_{DB}, is the temperature at which the ductile and brittle fracture stresses are equal. Below T_{DB} brittle fracture occurs, and above T_{DB} ductile fracture occurs. Increases in the strain rate or the presence of notches raises the local effective yield strength, the ductile fracture stress, and T_{DB} (Fig. 7.17). This model rationalizes the
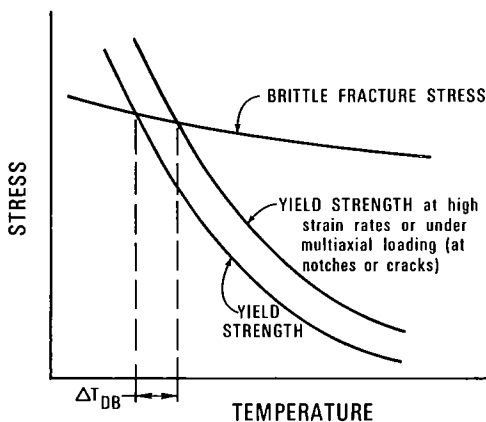
variability of the ductile–brittle transition temperature with material, strain rate, and other factors, but it cannot be verified experimentally, because under any set of experimental conditions only one of the two hypothesized fracture modes can be observed.

Ductile–brittle transitions occur in practically all b.c.c. materials, including iron, and in some h.c.p. materials. Face-centered cubic metals and alloys usually do not become brittle at low temperatures and have been considered less susceptible to brittle fracture than b.c.c. materials. Face-centered cubic metals that are precipitation hardened to very high strength levels may exhibit brittle behavior. Solution strengthening usually produces lower strength levels and more ductile behavior. Evidence for the toughness of copper and aluminum alloys has been reported by Reed and Mikesell (1967) and Kaufman and Johnson (1962). Details on the suitability of copper and aluminum alloys for use in toughness–sensitive applications may be found in Chapter 11, Structural Alloys. Fracture toughness has been found to vary inversely with yield strength within groups of closely related materials in several experimental studies (Wanhill, 1978; Hirth and Froes, 1977; Hahn and Rosenfield, 1975; Stout and Gerberich, 1978; Pellini, Goode, Puzak, Lange, and Huber, 1965; and Read and Reed, 1979, 1981). No quantitative theoretical explanation for this behavior has been found, although attempts have been made.

7.4.2 Fatigue

When materials are stressed repeatedly at loads below their ultimate tensile strengths, failure due to fatigue may eventually occur. Fatigue is discussed by Forrest (1966), Frost, Marsh, and Pook (1974), Kennedy (1963), and Sandor (1972). In standard texts, fatigue at low temperatures is treated briefly because fatigue strengths generally increase as the temperature is decreased and because of the large volume of room-temperature data available. Important exceptions are brittle materials, ductile or intermediate materials behaving in a brittle manner because of notches or stress concentrators, and ductile materials fatigued at constant strain amplitudes at high strains (low-cycle fatigue) (Nachtigall, Klima, and Freche, 1968). Fatigue testing of structural materials at low temperatures is done occasionally in cases where the adequacy of the fatigue resistance of a critical material is in question. Rotating superconducting machinery and pulsed superconducting magnets are examples of de-

Figure 7.17 Postulated temperature dependences of ductile and brittle fracture stresses showing effects of high strain rate and stress concentration.

vices that require low-temperature fatigue resistance.

Fatigue testing. The two common methods of applying fatigue stresses for testing are direct stress and rotating–bending. In direct stress, axial loads are repeatedly applied. In rotating–bending, rotation of the specimen or of a test fixture causes a bending moment to be applied repeatedly. Few experiments involving rotating–bending have been performed at cryogenic temperatures. Specimens for direct-stress fatigue testing closely resemble those used for tensile testing, i.e., smooth round bars with grips on the ends. Hourglass-like shapes are sometimes used to prevent buckling in compression. The testing machine for fatigue experiments must be capable of rapid cycling to accomplish tests in a reasonable amount of time. Two basic variations are used in controlling the deformation: load control and strain control. In load-controlled fatigue testing, the minimum and maximum loads on the specimen are held constant during cycling, regardless of the strain values obtained; conversely, in strain-controlled fatigue testing, the minimum and maximum strain values are controlled, regardless of the load values required. The distinction between these two types of control is significant when stresses above the yield strength are used because the relationship between stress and strain becomes nonlinear.

Results of a series of fatigue tests may be expressed as an S–N curve, in which the number of cycles to failure is plotted on a logarithmic x-axis, and the stress range is plotted on a linear y-axis. The endurance limit is the stress at which this curve becomes horizontal, if it does. If the curve does not become horizontal, the endurance limit is defined as the stress required for failure at a specified arbitrary number of cycles, such as 10^8.

Fatigue at low temperatures. Fatigue of pure metals at low temperatures was studied by McCammon and Rosenberg (1957). They found that in copper, silver, gold, aluminum, cadmium, and magnesium, the fatigue strength (cyclic-stress amplitude required to fracture the specimen in 10^5 cycles) increased significantly with decreasing temperature, roughly as the ultimate tensile strength. In zinc and iron, which are subject to a ductile–brittle transition, the fatigue strength was difficult to measure below the transition, because at low stresses the material appeared to last indefinitely, whereas at high

stresses, the material fractured in a brittle manner. The limited number of fatigue failures that were obtained in the midrange of stresses showed clearly that the fatigue strengths of these materials were decreased at temperatures below their ductile–brittle transitions. It was observed in this study that the increases observed in fatigue strength on cooling were due to increases in the resistance of the material to both crack initiation and crack growth.

Some fatigue data are available on structural materials at 4 K. Aluminum alloys were studied under load control by Schwartzberg, Kiefer, and Keys (1964). Aluminum and titanium alloys and steels were tested under strain control by Nachtigall et al. (1968) and Nachtigall (1975). At high strain levels fatigue resistance generally decreased with decreasing temperature; at low strain levels it increased.

The effect of the frequency of the applied stress on fatigue life is a difficult problem to study experimentally, although some work has been done at room temperature. The general conclusion from these studies is that increasing the frequency of stressing into the kilohertz range generally increases the endurance limit, with a maximum and subsequent decrease sometimes being observed. Very few data are available on this subject, which is not surprising in view of the experimental difficulties: accurate stress measurement over a wide frequency range requires unconventional techniques; heating from hysteresis increases with frequency; effects from corrosive environments may decrease with frequency. Changes in frequency, even well below the kilohertz range, can have pronounced effects on the rate of corrosion fatigue. In such situations, the fatigue life may decrease greatly with decreasing frequency. This can be a perplexing design problem because time for complete testing at low frequencies may not be available.

Cyclic strain at an amplitude of 1 to 2% and frequencies of about 0.3 Hz was found to produce heating in an AISI 304 specimen immersed in liquid helium (Shepic and Schwartzberg, 1978). This heating decreased at lower frequencies. It was implied that this frequency dependence of the specimen temperature, the cyclic stress–strain curve, and, presumably, the specimen fatigue resistance must be accounted for in 4-K fatigue testing.

7.4.3 Creep

Creep, that is, plastic deformation with time under constant load, is not significant in the de-

sign of most components for service at room and cryogenic temperatures. It could conceivably become important in the design of high-precision components for long service lives at high stresses (Wigley, 1971) or in components made from low-melting-point metals, such as indium O-rings and gaskets, and in polymers at and below room temperature. An investigation of creep in mercury and cadmium single crystals at liquid-helium temperatures was recently reported by Arko and Weertman (1969). It was established that creep is possible at liquid-helium temperatures, but that, contrary to the high-temperature behavior, stresses near the yield strength are required and creep rates decrease rapidly after loading.

7.5   ENGINEERING METALS AND ALLOYS

The behaviors of common engineering metals and alloys at low temperatures are described briefly to indicate the range of strength values produced by the various strengthening mechanisms in alloys based on several metallic elements. Because the mechanical properties of a given lot of material are determined by its own chemistry and processing, the data given below can only be exemplary. A detailed discussion of the relative advantages of candidate structural materials is presented in Chapter 11.

7.5.1   Copper and copper alloys

Room-temperature critical resolved shear strengths equivalent to a tensile yield strength of about 0.4 MPa (60 psi) have been reported for pure, well-annealed copper single crystals with very low dislocation densities (Young, 1968). This low value illustrates the fact that crystal-lattice defects in f.c.c. metals provide their strength; a material with practically no yield strength can be made by removing all dislocations, impurities, grain boundaries, and other strengthening lattice defects. Engineering materials are considerably stronger. More usual yield strength values for commercial purity copper are 20 to 60 MPa. Tensile yield and ultimate strengths as functions of temperature are shown in Fig. 7.18 for commercially pure (99.97% Cu) annealed copper; cold-drawn copper; admiralty brass, an alloy of copper with about 30 wt.% Zn, and a precipitation-hardened Cu–Be alloy. Behaviors typical of relatively pure, annealed f.c.c. metals as illustrated by 99.95% pure, annealed copper are: the low value of the yield strength at
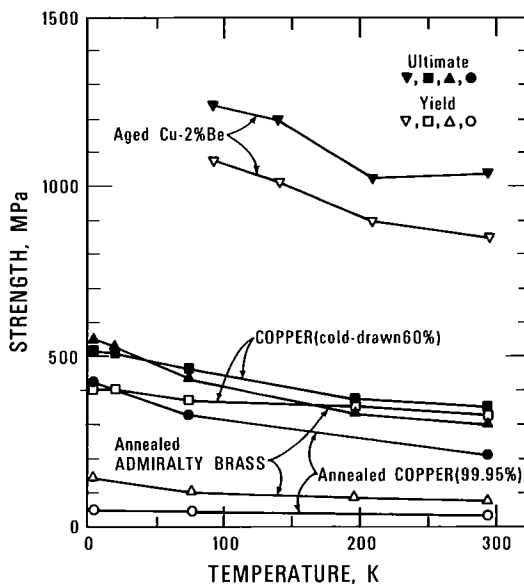

Figure 7.18 Yield and ultimate tensile strengths as a function of temperature for commercially pure, annealed, and cold-drawn copper (polycrystalline); annealed admiralty brass; and aged beryllium copper. Open symbols are yield strengths; closed symbols are ultimate tensile strengths (*Handbook on Materials for Superconducting Machinery*, 1977).

all temperatures, the weak dependence of yield strength on temperature, ultimate strength significantly larger than yield strength, and temperature dependence of the ultimate strength stronger than that of the yield strength. The large capacity of relatively pure f.c.c. metals for work hardening may be illustrated by a comparison of the behaviors of annealed and cold-drawn copper, also shown in Fig. 7.18. Cold drawing increases the yield strength to about 400 MPa and slightly increases the ultimate strength. The temperature dependence of the yield strength in the cold-drawn condition is weaker than in the annealed condition because work hardening increases the athermal component of the yield strength more than the thermal part in this material.

Up to about 33% of zinc is substitutionally soluble in copper. Above 33%, precipitates of the beta phase form, and ductility decreases at low temperatures. The f.c.c. solid solutions of zinc in copper are called the alpha brasses, the most useful class at low temperatures. Brass has been widely used for tubing and small machined parts in cryogenic equipment. The mechanical proper-

ties of a high-zinc alpha brass, admiralty brass, are compared with copper in Fig. 7.18. The brass has a higher yield strength by about a factor of 2 and a higher ultimate strength by about 40% over the range 0 to 300 K. The admiralty brass has a yield strength several times that shown in Fig. 7.8 for single crystal 70Cu–30Zn brass because of strengthening by grain boundaries, residual cold work, and additional chemical components. The temperature dependence of the yield strength of the commercial material is weaker than that of the single crystal because the additional strengthening mechanisms have raised the athermal part of the yield stress more than the thermal part. Most alpha brasses have sufficient toughness to prevent brittle fracture in low-temperature applications.

Alloys of copper with beryllium may be precipitation hardened. The strength properties of a typical alloy are also illustrated in Fig. 7.18. This graph shows the effectiveness of precipitation hardening in increasing strength. Usually, such dramatic increases in strength are accompanied by decreases in toughness, which limits the usefulness of some very strong materials in applications where toughness is important.

The low-temperature mechanical properties of copper and many copper alloys were compiled by Reed and Mikesell (1967).

7.5.2 Aluminum and aluminum alloys

Aluminum and its alloys exhibit much the same behavior as copper and its alloys. Observed yield strengths range from 0.5 MPa (70 psi) or less for the purest research material (99.999% Al single crystal) to well over 500 MPa for precipitation-hardened materials. The yield and ultimate strengths of three commercial alloys are compared in Fig. 7.19. Annealed commercial purity aluminum, alloy designation 1100-0, has a nearly constant yield strength of approximately 60 MPa over the range 0 to 300 K, and significant work hardening is shown by the considerable difference between yield and ultimate strengths. A common solution-strengthened aluminum alloy in the annealed condition, aluminum 5083-0, has yield and ultimate strengths over twice that of the 1100-0 alloy. The yield strength of a precipitation-strengthened aluminum alloy, aluminum 2014, is about 10 times that of the commercially pure material, and its ultimate strength is close to its yield strength. For the annealed solid-solution alloys 1100-0 and 5083-0, the yield strengths have weak temperature dependences and the ultimate
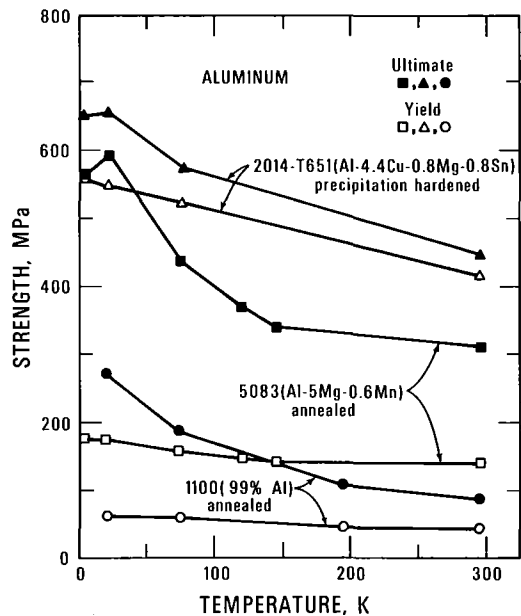

Figure 7.19 Yield and ultimate tensile strengths as a function of temperature for annealed, commercially pure, and solution-hardened aluminum and a precipitation-hardened alloy. Open symbols are yield strengths; closed symbols are ultimate tensile strengths (*Handbook on Materials for Superconducting Machinery*, 1977).

strengths have strong temperature dependences, behavior typical of f.c.c. materials. In contrast, the precipitation-hardened alloy has decreased capacity for work hardening. The difference of about a factor of 2 in the strength scales used for copper and aluminum alloys is explained by the difference of about the same factor in the shear moduli of these materials. Mechanical behavior of aluminum and dilute aluminum alloys has been reviewed by Reed (1972).

7.5.3 Austenitic stainless steels

Austenitic stainless steels are alloys of iron with chromium and enough nickel or manganese, or both, to ensure the stability of the f.c.c. (austenitic) crystal structure at room temperature. Other constituents, especially carbon and nitrogen, significantly affect the behavior of these steels and the stability of the austenite at low temperatures. The austenitic phase is metastable in most of these alloys and martensitic transformations may occur on cooling to cryogenic temperatures or during deformation at low temperatures. The transformation products influence the strength, toughness, and deformation

behavior, as discussed in Chapters 9 and 11. Austenitic stainless steels are discussed extensively in the *Handbook of Stainless Steels* (1977).

The strength of annealed austenitic stainless steels is produced primarily by solution hardening. These steels work-harden significantly; therefore thermal and mechanical treatments significantly affect their mechanical properties.

Nitrogen additions significantly increase both the room- and low-temperature strengths of austenitic stainless steels. For example, increasing the nitrogen content 0.1% can double the 4-K yield strength of 19Cr–9Ni stainless steel (Read and Reed, 1979). Norstrom (1977) has shown that the nitrogen appears to have two separate effects. It provides solution hardening, which is especially effective in raising the low-temperature strength, and it significantly enhances the hardening effect of the grain boundaries, providing strength increases at moderately high as well as at low temperatures. This is an example of the complexity of strengthening mechanisms in practical materials.

Strengths of several versions of 19Cr–9Ni stainless steel (AISI type 304) are plotted against temperature in Fig. 7.20. It can be seen that substantial increases in yield strengths are obtainable by work hardening and by adding nitrogen. Stainless steel AISI 304N contains intentionally added nitrogen (0.12 wt.% for the material of Fig. 7.20), which greatly increases the low-temperature yield strength. The general level of strengths available is higher than for aluminum or copper alloys because of the higher shear modulus of stainless steel.

The strain-hardening behavior of austenitic stainless steels at low temperatures is influenced strongly by deformation-included martensitic transformations. The 4-K stress–strain curves of Guntner and Reed (1962) for AISI 304, which forms deformation-induced martensites at 4 K, and AISI 310, which does not, show that the strain-hardening coefficient of the AISI 304 is more than 5 times larger than that of AISI 310 at strains of 15 to 30%.

Fatigue test results for AISI 304L (low carbon) and 304N stainless steel are shown in Fig. 7.21 (Nachtigall, 1975; Shepic and Schwartzberg, 1978). Note that the fatigue resistance at liquid-nitrogen and liquid-helium temperatures is equal or slightly better than that at room temperature. A slight extrapolation of Nachtigall's results to 10^6 cycles indicates that the cyclic stress range required for failure at 10^6 cycles is of
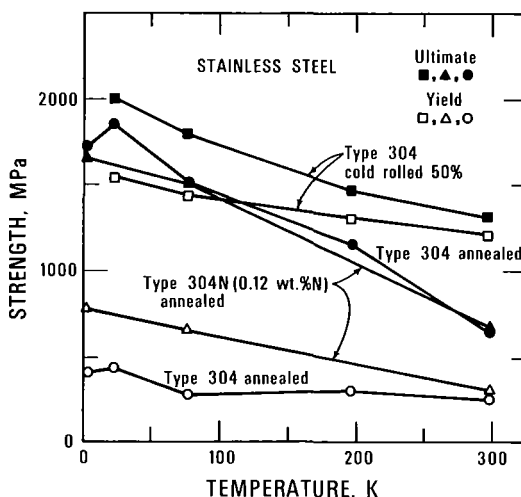

Figure 7.20 Yield and ultimate tensile strengths as a function of temperature for annealed, nitrogen-strengthened, and cold-drawn stainless steel 304. Open symbols indicate yield strengths; closed symbols indicate ultimate tensile strengths (*Handbook on Materials for Superconducting Machinery*, 1977; Read and Reed, 1979).
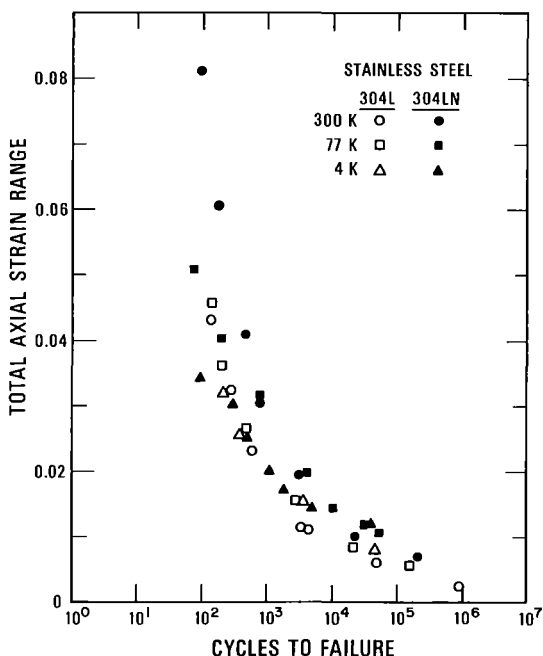

Figure 7.21 Fatigue test results for stainless steels 304L and 304LN at 300, 78, and 4 K (Nachtigall, 1975; Shepic and Schwartzberg, 1978).

the order of half the ultimate tensile stress, for fatigue at zero mean stress. Total stress ranges of around 800 MPa (116 ksi) were required to produce failure of Nachtigall's stainless steel 304L specimens after 10^6 cycles.

7.5.4 Titanium and titanium alloys

Titanium alloys, used in aerospace applications because of their low density and high strength at high temperatures, are also useful at low temperatures because of their high strength and low thermal conductivity. The toughness of some titanium alloys decreases severely at temperatures in the neighborhood of 77 K and below, limiting their usefulness at lower temperatures. Strengths of some commercial titanium alloys are shown in Fig. 7.22.

The crystal structure of titanium alloys is variable through their chemical compositions and thermal and mechanical treatments. Titanium-based materials potentially useful for cryogenic structures include unalloyed titanium, Ti–5Al–2.5Sn, and Ti–6Al–4V. Unalloyed titanium

and Ti–5Al–2.5Sn have the h.c.p. crystal structure (referred to as the α phase in this context), and Ti–6Al–4V contains precipitates of the b.c.c. structure (Ti–6Al–4V is said to contain α and β phases). Interstitial carbon, oxygen, and nitrogen in small amounts can severely embrittle titanium alloys at low temperatures (Shannon and Brown, 1968; Tobler, 1976b). The annealed commercial titanium alloys have only slight work-hardening capacity compared with annealed austenitic stainless steels. Titanium alloy ultimate strengths range from about 1.25 times the yield strength for commercially pure titanium at room temperature to 1.03 times the yield strength for precipitation-hardened titanium alloy (Ti–6Al–4V) at 4 K; their ultimate strengths have slightly weaker temperature dependences than the yield strengths. The high, strongly temperature-dependent yield strengths of these materials and the relatively small and constant differences between their yield and ultimate strengths are due to their mainly h.c.p. crystal structure. More highly alloyed materials, with a higher β phase (b.c.c.) content, exhibit these same behaviors.

7.5.5 Iron and b.c.c. iron alloys

Iron is by far the most widely used metallic element in structural metals and alloys, but the low-temperature ductile–brittle transition (see § 7.4.1) of many ferrous alloys limits their usefulness at low temperatures. Typical b.c.c. behavior is demonstrated most clearly by the pure iron and carbon steels. The yield strength is high even in pure iron and has a strong temperature dependence (Fig. 7.23). The ultimate strength is only slightly above the yield strength at room temperature and approaches the yield strength with decreasing temperature. The tensile properties of the carbon steel give fair warning of its low toughness at low temperatures: the yield and ultimate strengths are practically identical and the elongation and reduction of area are low. Addition of nickel lowers the ductile–brittle temperature and increases the elongation at low temperatures (Fig. 7.24), making Fe–9Ni, for example, useful at temperatures as low as 77 K, if it is properly processed. The addition of nickel (f.c.c.) to iron (b.c.c.) increases the austenite stability, providing some retention of austenite at lower temperatures. The presence of the austenite at lower temperatures is thought to contribute to greater ductility, effectively serving to lower the ductile–brittle transition temperature.
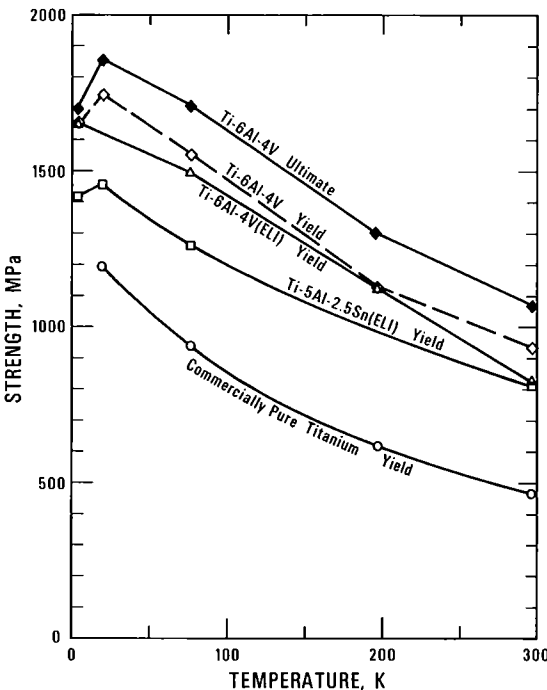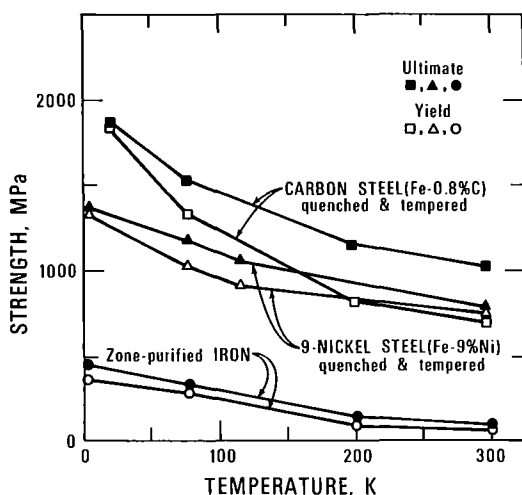

Figure 7.22 Yield strengths as a function of temperature for titanium, Ti-5Al-2.5Sn (ELI), Ti-6Al-4V (ELI), and Ti-6Al-4V, and ultimate strength of Ti-6Al-4V (*Handbook on Materials for Superconducting Machinery*, 1977, and Tobler, 1976a).

Figure 7.23 Yield and ultimate tensile strengths as a function of temperature for zone-purified iron, 9-nickel steel, and carbon steel. Open symbols indicate yield strengths; closed symbols indicate ultimate strengths (Smith and Rutherford, 1957; Tobler, 1976a; Warren and Reed, 1963).
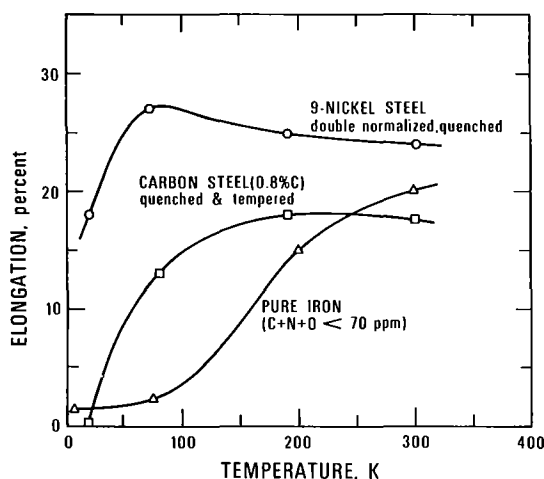

Figure 7.24 Elongation to fracture for zone-purified iron (elongation only is shown), 9-nickel steel, and carbon steel plotted against temperature (Smith and Rutherford, 1957; Tobler, 1976a; Warren and Reed, 1963).

7.6 POLYMERS AND GLASSES
7.6.1 Polymers

Polymers exhibit strikingly varied behavior depending on the temperature and the time scale of the observation. A classic simple polymer observed for common experimental times of the order of 1 s to 1 h has a glassy region, a rubbery

region, and a viscofluid region as the temperature is raised. These behaviors are demonstrated by loading a polymer specimen to a given stress, measuring the magnitude of the deformation produced after a fixed interval of time, and plotting deformation as a function of temperature, as shown in Fig. 7.25 for an amorphous polymer. The term *glassy* indicates the similarity observed between the properties of polymers at low temperatures and those of glass. As the temperature is raised, the polymer becomes less brittle, and its strength and moduli begin to drop steadily and significantly. Between the glassy and rubbery regions, the polymer passes through a leathery region. Throughout the leathery region, the polymer exhibits some ductility and toughness, along with rapidly decreasing strength and moduli. Eventually the behavior stabilizes in the rubbery region, where the polymer can be stretched severely and will eventually return almost to its original size. The deformation in the rubbery region is strongly suppressed in a crystalline polymer (discussed below).

As the temperature is raised further, the polymer eventually becomes a viscous liquid, then less viscous, and then melts completely.

The glass transition temperature, T_g, is the approximate upper limit of the glassy region. It is usually determined experimentally by a thermal expansion technique. Commonly observed values of T_g are in the range 220 to 370 K. Glassy mechanical behavior is not restricted to temperatures below T_g. High values of the elastic shear modulus can be observed at temperatures above T_g if stresses and strains are applied and measured within short times. Significant plastic deformation can be obtained in tensile stress-strain experiments below T_g, even at conventional rates of deformation (see "Amorphous and crystalline polymers," below).
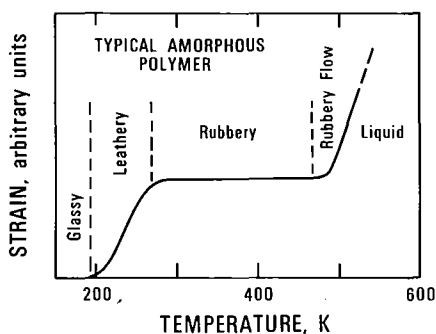

Figure 7.25 Thermomechanical curve of a simple polymer.

In polymers, a distinction must be made between nonlinear deformation, that is, lack of a linear relationship between stress and strain; viscoelastic behavior, in which strain increases with time under a constant stress; and irreversible plastic flow. Nonlinear viscoelastic strains can be almost completely recovered in the rubbery behavior region. But in the glassy region, a nearly linear portion of the stress–strain curve is followed by nonrecoverable strain at nearly constant load. Just below T_g, strains of 300 to 1000% may be obtained (Ward, 1971). Well below T_g, rupture occurs at low strains ($\sim 10\%$).

Recoverable deformation in polymers occurs by the mechanism of thermally activated reorientation of molecular chain segments toward the stress axis (Meares, 1967). Nonrecoverable deformation has contributions from this mechanism and additional contributions from molecular chains sliding past one another. This sliding is made possible by the severing of some molecular links.

An extensive compilation of the mechanical properties of some polymers including polytetrafluoroethylene (TFE), polyethylene terephthalate (PET), polycarbonate (PC), and four others, has been prepared by Schramm, Clark, and Reed (1973). The low-temperature strengths of polymers are well below those of the usual structural metals. The temperature dependences of their tensile strength and elongation are usually significant, as in the b.c.c. metals. The tensile strengths of polychlorotrifluoroethylene (CTFE), PET, PC, TFE, and an epoxy resin (NASA Resin 2) are shown in Fig. 7.26.

Amorphous and crystalline polymers.

Polymers exist in amorphous and crystalline states. The crystalline state in a polymer means that the molecules occupy positions of three-dimensional order. The degree of crystallinity of a specimen of a crystallizable polymer is determined by the time–temperature path followed on cooling from the molten state. In some cases, minute single crystals have been obtained by crystallization from very dilute solutions. In PET, stretching an amorphous specimen produces small crystallites oriented along the stress axis.

Crystallinity in polymers has profound effects on their mechanical properties. Deformability in the glassy temperature region is suppressed to such a degree that they may become quite brittle below T_g, in contrast to amorphous polymers, which generally can sustain some forced defor-
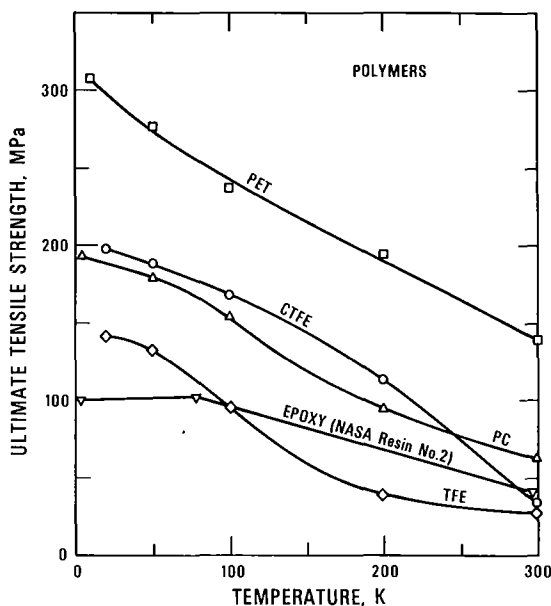
Figure 7.26 Ultimate tensile strengths as a function of temperature for CTFE, PET, PC (Schramm et al., 1973), and NASA Resin 2 (Soffer and Molho, 1967).

mation at and slightly below T_g. The strain produced by a given stress level in the leathery and rubbery temperature regions is likely to be much lower in the crystalline state than in the amorphous state. Thus, increased crystallinity in a polymer increases its strength, but also promotes brittle behavior at temperatures below T_g.

A dramatic instance of the effect of increased crystallinity on strength has been reported by Armeniades, Kuriyama, Roe, and Baer (1968). They found that the tensile strength of 54% crystalline PET film was more than double that of amorphous PET film over the temperature range 110 to 300 K. Even larger differences were reported below 110 K. Differences in Young's modulus of more than a factor of 10 between natural rubber specimens of about 5 and 20% crystallinity are described by Meares (1967).

Crazing.

Crazes, which are opaque striations in planes normal to the tensile stress, form in some amorphous polymers, notably polymethylmethacrylate and polystyrene during plastic deformation in the brittle temperature range (Ward, 1971). Crazes can also be formed by chemical action. Research into the structure of crazes has shown them to be regions of lower refractive index and lower density than the surrounding material (Berry, 1972). These property

changes are due to microscopic voids. It was found that crazes in PMMA contained 50 to 55% by volume of free space, presumably in the form of interconnecting voids. Crazes are sharply bounded; their thickness is much less than their other dimensions. They have significant load-bearing capacity, although they do not have the strength of the surrounding material.

Crazes and the submicroscopic flaws from which they grow control the tensile strength of polymers in the range of brittle behavior, which is a large portion of the cryogenic temperature range for most polymers. The existence of these intrinsic, so far undetectable, flaws is demonstrated by measurements of the tensile strength of specimens with externally introduced flaws of varying sizes. The data fit the Griffith theory of brittle fracture (Chapter 8) for externally introduced flaws down to a certain size, a_0. Smaller, externally introduced flaws have no effect on the strength, indicating the existence of intrinsic flaws of size a_0, which control the tensile strength of the polymer (Berry, 1972). At sufficiently high stresses, these invisible intrinsic flaws become visible crazes; fracture then follows, initiating at one of the crazes. In a study on polymethylmethacrylate (PMMA), the crazing was found to be much more severe and the effective intrinsic flaw size over twice as large at liquid-nitrogen temperature than at room temperature. Data on the relationship between crazing, flaw size, and tensile strength for other polymers are less conclusive, but similar effects to those observed in PMMA cannot be ruled out (Berry, 1972).

7.6.2 Glass

"Glass can be defined as an inorganic product of fusion which has been cooled to a rigid condition without crystallization" (Jastrzebski, 1959). Glasses have an amorphous structure characteristic of a supercooled liquid made up of a three-dimensional network of primary units, which have local order but no long-range order (Zarzycki, 1968). The lack of long-range crystalline order in glasses precludes the formation and motion of dislocations; therefore plastic deformation by dislocation motion is absent. At high temperatures (800 K and above), ordinary glasses behave as viscous liquids, flowing gradually under applied stress (Babcock, 1977). At room temperature and below, however, the viscosity of glass is so high that it behaves as a brittle solid, because no mechanism of plastic deformation on a practical time scale is available.

The theoretical strengths of glasses are of the order of 10 GPa (Phillips, 1972). These strengths have been achieved in practice in small samples under carefully controlled laboratory conditions. In most standard tests of a group of glass specimens, the failure stresses have been found to be widely distributed, with the minimum found to be of the order of 35 MPa (Hillig, 1961). This is due to the presence of stress concentrators on the surfaces of virtually all bulk glasses. The inability of glass to deform plastically prevents relief of the stresses around such flaws, and brittle fracture results. Glass is quite strong in compression, and the cause of specimen failure in compression tests is believed to be a tensile component of the stress that could not be eliminated by the experimental arrangement.

Kropschot and Mikesell (1957) and Hillig (1961) report increases in the strength of glasses on cooling from room to liquid-nitrogen temperature, but no further improvement was observed in tests conducted at 20 K. Like the room-temperature strength, the practical low-temperature strength of glass is far lower than the theoretical value because of fracture initiation at surface flaws.

Fatigue damage in the conventional sense does not occur in glass, but the application of a constant stress for a long time may result in failure. This is known as static fatigue and is believed to be caused by the growth of preexisting flaws to critical dimensions under the action of a steady stress. Fluctuating stress may also produce flaw growth at about the same rate as an equivalent static stress.

Glass may be fractured by thermal stresses because of its low thermal conductivity, which results in nonuniform contraction and, therefore, internal stresses under rapid cooling. This liability can be mitigated by specifically designing the chemical composition of the glass to produce low thermal contraction.

7.7 SOURCES OF DATA

Many compilations and handbooks of low-temperature mechanical properties exist. Some examples are the *Handbook of Materials for Superconducting Machinery* (1977) maintained by the Metals and Ceramics Information Center, Battelle Columbus Laboratories, and the handbook entitled *LNG Materials and Fluids* (1977) by the National Bureau of Standards. *Mechanical Properties of Materials at Low Temperatures* by Wigley (1971) contains property information,

guidance on material selection, and many references.

Proceedings of topical conferences on cryogenic materials are useful data sources. These include the biennial publication *Advances in Cryogenic Engineering*, now in its twenty-eighth volume, which contains proceedings from the Cryogenic Engineering Conference and the International Cryogenic Materials Conference. The International Cryogenic Materials Conference has recently started a new series of conference proceedings, the Cryogenic Materials series, beginning with *Nonmetallic Materials and Composites at Low Temperatures* (1979). The American Society for Testing and Materials in Philadelphia, Pennsylvania, has sponsored recent conferences on cryogenic materials and published the proceedings as ASTM special technical publications, *Fatigue and Fracture Toughness—Cryogenic Behavior* (1974) and *Properties of Materials for Liquefied Natural Gas Tankage* (1975).

The *Metals Abstracts* indexes data on the mechanical properties of metals and alloys from journals and reports. Wohlbier's annual mechanical properties volumes (Wohlbier, 1974; 1975; 1976) provide useful summaries of current mechanical property literature. A critical survey of data sources on mechanical properties of metals has been compiled by Gavert, Moore, and Westbrook (1974).

References that provide background material on and more detailed discussions of topics covered in this chapter are listed in Table 7.2.

7.8 AUTHOR'S AFTERWORD

The mechanical behaviors of metals at low temperatures are generally well understood on physical grounds, measurement techniques are adequate to determine the necessary material properties, and a substantial amount of test data on practical materials has been accumulated. Opportunities for new knowledge and practical advantages remain. Solution softening in b.c.c. materials and low-temperature anomalies of the temperature dependence of the flow strengths of some f.c.c. materials are not well understood. The often-observed inverse correlation between strength and toughness lacks a quantitative explanation. Stronger and cheaper welds with adequate toughness for low-temperature service are needed. More convenient and economical low-temperature test techniques, especially for toughness, would be useful.

Table 7.2   References on background material for low-temperature mechanical properties of materials.

§ 7.2	Temperature dependence of strengthening and flow
	Arsenault (1975)
	Conrad (1960, 1964)
	Cottrell (1956)
	Li (1968)
	McLean (1962)
	Nix and Menezes (1970)
	Peckner (1964)
	Reed-Hill (1964)
	Tegart (1966)
	Tien and Ansell (1966)
	Weertman and Weertman (1965)
§ 7.2.1	Basic mechanism of plastic flow in crystalline solids
	Friedel (1967)
	Gilman and Johnston (1967)
	Johnston and Gilman (1959)
	Nabarro (1967)
	Read (1953)
§ 7.2.3	Crystal structure effects
	Christian (1970)
	Hirsch (1968)
	Soo and Galligan (1969)
	Stein and Low (1966)
§ 7.2.5	Solution strengthening
	Haasen (1965)
	Haasen (1968)
	Fleischer and Hibbard (1963)
§ 7.2.6	Athermal strengthening by grain boundaries and precipitates
	Brown and Ham (1971)
§ 7.3.3	Martensitic transformations
	Reed (1962)
§ 7.3.5	Yield points
	Hahn (1962)
§ 7.4.2	Fatigue
	Forrest (1966)
§ 7.6.1	Polymers
	Argon (1973)
	Billmeyer (1962)
	Bueche (1962)
	Meares (1967)

The austenitic stainless steels have been used for years where strength and toughness at low temperatures are required. These materials have been chosen recently for use in the Mirror Fusion Test Facility at Lawrence Livermore Laboratory and in two of the three United States–manufactured magnets for the Large Coil Project at Oak Ridge National Laboratory. The strength of the austenitic stainless steels can be significantly increased by adding nitrogen, although this decreases the toughness; thus an alloy can be custom-made for an application requiring a specific strength level. Possible improvements in

the characteristics of these alloys through manganese additions are currently under study. A serviceable material for most low-temperature structural applications can be found among the austenitic stainless steels.

Because cryogenic engineering on the structural size scale of the current fusion magnets is a new field, a service history for austenitic stainless steels needs to be established. Construction materials should be carefully characterized; structures should be thoroughly instrumented; and detailed performance records should be kept and made generally available to designers. Only through such efforts will the durability and economy of future cryogenic structures be ensured.

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