Designation: E1450 – 16

Standard Test Method for Tension Testing of Structural Alloys in Liquid Helium[1]

This standard is issued under the fixed designation E1450; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes procedures for the tension testing of structural alloys in liquid helium. The format is similar to that of other ASTM tension test standards, but the contents include modifications for cryogenic testing which requires special apparatus, smaller specimens, and concern for serrated yielding, adiabatic heating, and strain-rate effects.

1.2 To conduct a tension test by this standard, the specimen in a tensile cryostat is fully submerged in normal liquid helium (He I) and tested using crosshead displacement control at a nominal strain rate of 10^{-3} mm/mm/s or less. Tests using force control or high strain rates are not considered.

1.3 This standard specifies methods for the measurement of yield strength, tensile strength, elongation, and reduction of area. The determination of the Young's modulus is treated in Test Method E111.

NOTE 1—The boiling point of normal liquid helium (He I) at sea level is 4.2 K (−269°C or −452.1°F or 7.6°R). It decreases with geographic elevation and is 4.0 K (−269.2°C or −452.5°F or 7.2°R) at the National Institute of Standards and Technology in Colorado, 1677 m (5500 ft) above sea level. In this standard the temperature is designated 4 K.

1.4 Values stated in SI units are treated as primary. Values stated in U.S. customary units are treated as secondary.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* See Section 5.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

A370 Test Methods and Definitions for Mechanical Testing of Steel Products

E4 Practices for Force Verification of Testing Machines

E6 Terminology Relating to Methods of Mechanical Testing

E8/E8M Test Methods for Tension Testing of Metallic Materials

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E83 Practice for Verification and Classification of Extensometer Systems

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E111 Test Method for Young's Modulus, Tangent Modulus, and Chord Modulus

E1012 Practice for Verification of Testing Frame and Specimen Alignment Under Tensile and Compressive Axial Force Application

3. Terminology

3.1 *Definitions of Terms Common to Mechanical Testing*—

3.1.1 The definitions of mechanical testing terms that appear in the Terminology E6 apply to this test method. These terms include bending strain, elongation, extensometer, force, gauge length, proportional limit, reduced section, reduction of area, stress-strain diagram, tensile strength, and Young's modulus.

3.1.2 In addition, the following common terms from Terminology E6 are defined:

3.1.3 *adjusted length of the reduced section*—the length of the reduced section plus an amount calculated to compensate for strain in the fillet region.

3.1.4 *discontinuous yielding, n*—in a uniaxial test, a hesitation or fluctuation of force observed at the onset of plastic deformation, due to localized yielding.

3.1.4.1 *Discussion*—The stress-strain curve need not appear to be discontinuous.

3.1.5 *discontinuous yielding stress, σ_i*—the peak stress at the initiation of the first measurable serration on the curve of stress-versus-strain.

3.1.5.1 *Discussion*—The parameter σ_i is a function of test variables and is not a material constant.

[1] This test method is under the jurisdiction of ASTM Committee E28 on Mechanical Testing and is the direct responsibility of Subcommittee E28.04 on Uniaxial Testing.

Current edition approved Nov. 15, 2016. Published February 2017. Originally approved in 1992. Last previous edition approved in 2009 as E1450 – 09. DOI: 10.1520/E1450-16.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3.1.6 *gauge length, n*—the original length of that portion of the specimen over which strain, elongation, or change of length is determined.

3.1.6.1 *Discussion*—Typically, this length is also the distance between gauge marks, if gauge marking is used to facilitate measurement of the elongation after fracture.

3.1.6.2 *Discussion*—When sensing extension or motion with a gauge length that is predetermined by the specimen geometry or specific test method, then only resolution and strain error for the specified gauge length should determine the class of the extensometer system.

3.1.7 *length of the reduced section*—the distance between the tangent points of the fillets that bound the reduced section.

3.1.8 *reduced section*—the central portion of the specimen that has a cross section smaller than the gripped ends.

3.1.8.1 *Discussion*—The cross section is uniform within prescribed tolerances.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *adiabatic heating*—the internal heating of a specimen resulting from tension testing under conditions such that the heat generated by plastic work cannot be quickly dissipated to the surrounding cryogen.

3.2.2 *Dewar*—a vacuum-insulated container for cryogenic fluids.

3.2.3 *tensile cryostat*—a test apparatus for applying tensile forces to test specimens in cryogenic environments Fig. 1.

4. Significance and Use

4.1 Tension tests provide information on the strength and ductility of materials under uniaxial tensile stresses. This information may be useful for alloy development, comparison and selection of materials, and quality control. Under certain circumstances, the information may also be useful for design.

4.2 The force-time and force-extension records for some alloys tested in liquid helium using displacement control are often serrated (**1**).[3] Serrations are formed by repeated bursts of unstable plastic flow and arrests. The unstable plastic flow (discontinuous yielding) is a free-running process occurring in localized regions of the reduced section at higher than nominal rates of strain with internal specimen heating. Examples of serrated stress-strain curves for a typical austenitic stainless steel with discontinuous yielding are shown in Fig. 2.

4.3 A constant specimen temperature cannot be maintained at all times during tests in liquid helium. The specimen temperature at local regions in the reduced section rises temporarily above 4 K during each discontinuous yielding event (see Fig. 2), owing to adiabatic heating. The number of events and the magnitude of the associated drops in magnitude of force are a function of the material composition and other factors such as specimen size and test speed. Typically, altering the mechanical test variables can modify but not eliminate the discontinuous yielding (**2-4**). Therefore, tensile property measurements of alloys in liquid helium (especially tensile strength, elongation, and reduction of area) lack the usual significance of property measurements at room temperature where deformation is more nearly isothermal and discontinuous yielding typically does not occur.

4.4 The stress-strain response of a material tested in liquid helium depends on whether force control or displacement

[3] The boldface numbers in parentheses refer to the list of references at the end of this test method.

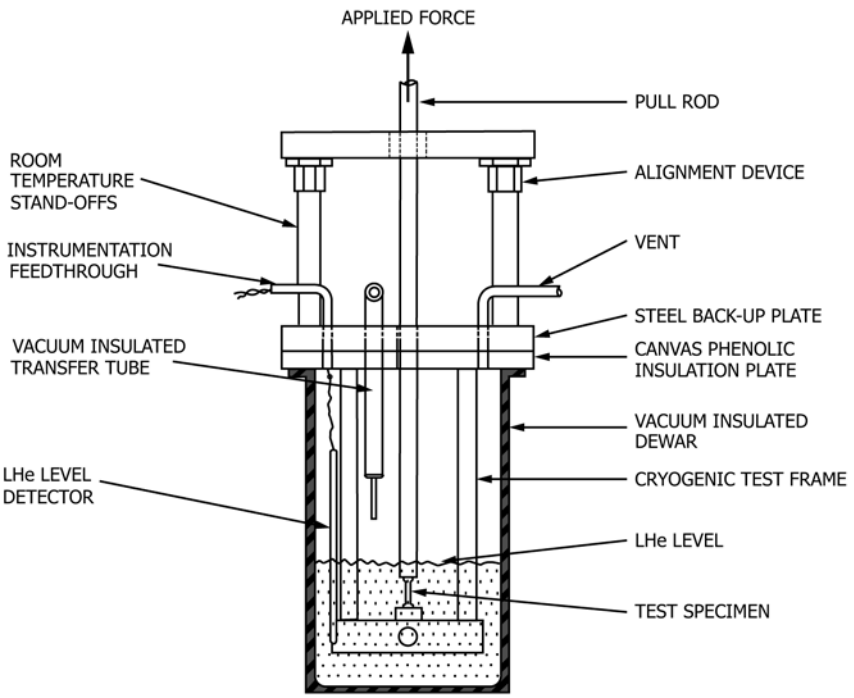


FIG. 1 Schematic Illustration of Typical Tensile Cryostat for Tension Testing at 4 K

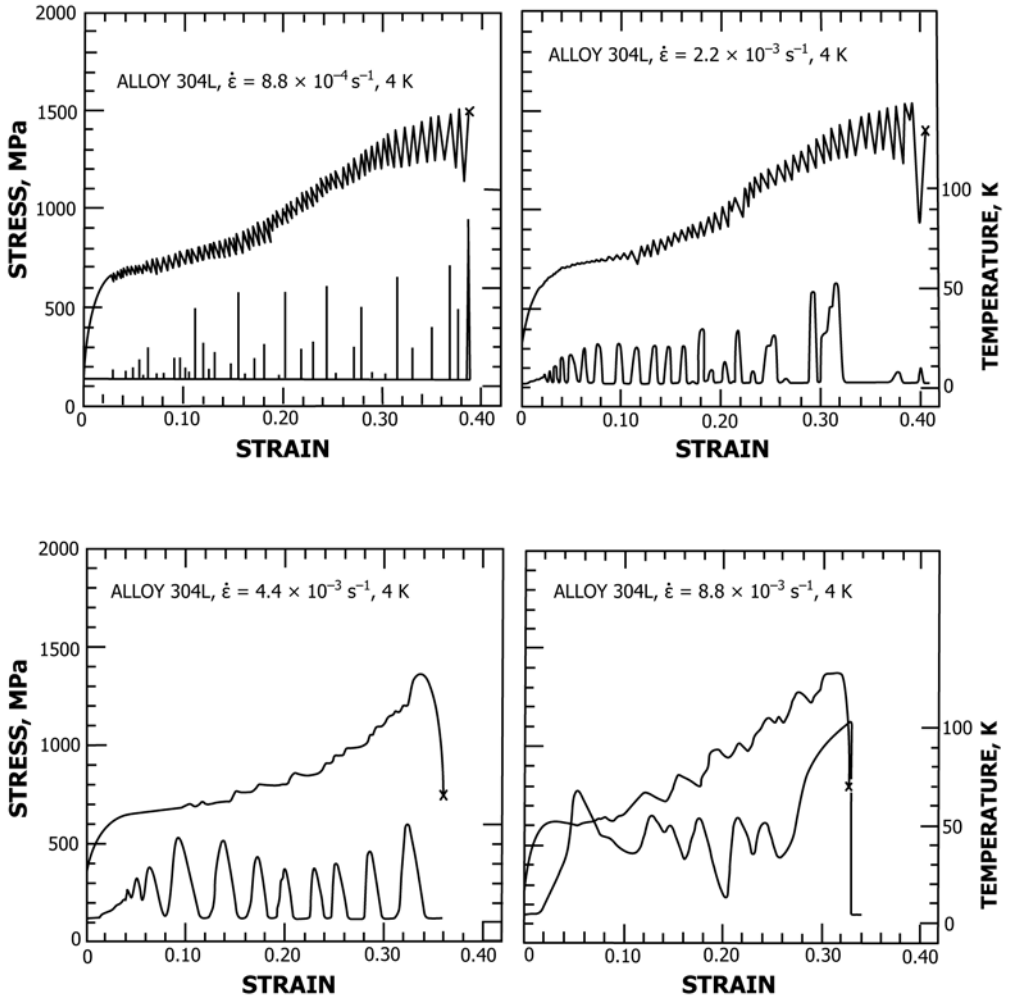


FIG. 2 Typical Engineering Stress-Strain Curves and Specimen Temperature Histories, at Four Different Nominal Strain Rates, for AISI 304L Stainless Steel Tested in Liquid Helium (4)

control is used (**3**). Crosshead displacement control is specified in this standard since the goal is material characterization by conventional methods. The possibility of a different and less favorable material response must be taken into account when data are used for design in actual applications subject to force-controlled conditions.

5. Hazards

5.1 Several precautions must be observed in the use of cryogenic fluids and equipment. Skin or eye contact with cryogens will freeze and destroy tissue. The appropriate protection may require goggles, clothing without pockets or cuffs, gloves, and tongs for handling cold specimens. Cryogenic containers that are internally pressurized or evacuated are potentially hazardous in that damage or leaks can produce explosions or implosions. Also, when liquids evaporate to gases, there is a huge volume increase; therefore asphyxiation is a potential threat where liquid nitrogen or liquid helium evaporates in rooms that are not properly ventilated. Safety guidelines pertaining to the use of liquid helium and other cryogenic fluids are considered elsewhere in more detail (**5**).

6. Apparatus

6.1 *Test Machines*—Use a test machine that meets the requirements of Practices E4 regarding verification of force accuracy.

NOTE 2—Because it is important to minimize heat loss from the dewar through the cryogenic test frame (Fig. 1), the cross-sections of these components are often smaller than they would be in a conventional test machine. A drawback to these smaller cross sections is that the compliance of the test frame, (displacement per unit of applied force), can be unacceptably large. High-compliance test frames can introduce artifacts in the stress-strain curve that complicate the interpretation of discontinuous yielding. It is often useful to characterize the compliance of the test frame before use. Measure the compliance by coupling the force train without including a specimen, by replacing the specimen with a rigid block, or by using a special calibration specimen. Then, measure the compliance at a low force and at the highest force expected in use.

6.2 *System Design*—The apparatus may be designed to accommodate one of the small specimens cited in 8.2.1 of this test method.

NOTE 3—2 Typically, alloys in liquid helium exhibit double or triple their ambient strengths. For the same specimen geometry, higher forces must be applied to the tensile cryostat, test specimen, force train members,

and grips at cryogenic temperatures. Many conventional test machines have a maximum force of 100 kN (22 480 lbf) , which may be insufficient for testing full-size specimens.

6.3 *Construction Materials*—To prevent service failures, fabricate the grips and other force-train members using strong, tough, cryogenic alloys.

NOTE 4—Many construction materials, including the vast majority of ferritic steels, are brittle at 4 K. Materials that have low thermal conductivity are desirable to reduce heat flow. Austenitic stainless steels (AISI 304LN), maraging steels (200, 250, or 300 grades, with nickel plating to prevent rust), and extra-low-interstitial (ELI) grade titanium alloys (Ti-6Al-4V and Ti-5Al-2.5Sn) have been used with proper design, for grips, pull rods, and tensile cryostat frames. Nonmetallic materials (for example, glass-epoxy composites) are excellent insulators and are sometimes used for compression members.

6.4 *Alignment:*

6.4.1 Single- and multiple-specimen systems shall meet Practice E1012 Class 10 alignment at room temperature.

NOTE 5—Proper system alignment is essential to avoid bending strains in the tension tests. This requirement will minimize contributions from the test apparatus to the bending strain. Tests performed with a qualified apparatus may still vary in amount of bending strain owing to small variations in the proposed test specimen configurations, or differences in machining.

6.5 *Gripping Mechanisms*—The choice of gripping mechanism to be used is influenced by specimen type. The mechanisms described in Test Methods E8/E8M are satisfactory at 4 K, but cryogenic materials shall be used in the construction of components to avoid failure in service.

6.6 *Dimension-Measuring Devices*—For measuring the dimensions of specimens, use a micrometer or other device that is accurate and precise to at least one-half of the smallest unit to be measured.

6.7 *Tensile Cryostats and Support Apparatus:*

6.7.1 *Tensile Cryostats*—The tensile cryostat may employ adjustable force-columns to facilitate alignment. A Dewar capable of retaining liquid helium is required.

NOTE 6—In general, tensile cryostat force-application frames for existing test machines are custom-built, but they may accommodate commercially available Dewars. Several practical designs, including turret-disc designs for multiple-specimen testing with a single cooling, are discussed in Refs (**6-10**). Stainless steel Dewars are safer (that is, more fracture resistant) than glass Dewars and less expensive than fiberglass Dewars. Generally, a single helium Dewar (see Fig. 1) is sufficient for short-term tensile tests. Also possible is a double-Dewar arrangement in which an outer Dewar of liquid nitrogen surrounds the inner Dewar of liquid helium.

6.7.2 *Ancillary Equipment*—Dewars and transfer lines for liquid helium must be vacuum insulated. Vacuum pumps, pressurized gas, and liquid nitrogen facilities are therefore required. After testing, the helium may be released to the atmosphere (see Section 5), recycled as a gas, or reliquefied.

NOTE 7—Recycling or reliquefaction requires large investments in purification and support systems.

6.8 *Temperature Maintenance and Liquid-Level Indicators*—Ensure that specimen remains fully submerged in liquid helium during the test.

NOTE 8—When the specimen is completely immersed, a simple indicator or meter, instead of a thermocouple can ensure that the specimen remains fully submerged throughout the test. An on-off indicator of the carbon-resistor type located at some reference point in the tensile cryostat can be used to verify that the liquid level always remains above the specimen. Alternatively, the liquid level can be continuously monitored using a superconducting wire sensor of appropriate length positioned vertically inside the tensile cryostat.

6.9 *Axial Strain Measurement:*

6.9.1 *Strain-Averaging Technique*—Nonaxiality of applied force (which may be introduced due to the machining of the test specimens) is usually sufficient to introduce errors in tension tests at small strains when strain is measured at only one position on the specimen. Therefore measure strains at three equally spaced (or, if good alignment has been achieved, at least two opposing) positions within the reduced section. Report the average of the strains from the two or three positions centered on the reduced section.

6.9.2 *Strain Gages:*

6.9.2.1 *Precautions*—Strain-gage films bonded directly to the specimen surface may be used to measure strain at 4 K (**11**).

NOTE 9—The use of bonded strain gages at 4 K, however, requires precautions not customarily required at room temperature. There are two major complications: the gage factor varies with temperature, and thermal output (apparent strain) is introduced as the specimen-gage combination is cooled from room temperature to 4 K. Thermal output is caused by two concurrent and algebraically additive effects in the strain gage installation: *(1)* the electrical resistivity of the gage grid element and *(2)* the differential thermal expansion between the gage grid element and the test specimen to which the gage is bonded. Failure to account for these effects can introduce considerable error in strain measurements.

NOTE 10—Gage manufacturers generally do not supply thermal output data at 4 K; neither do they state gage factors at 4 K. For high accuracy the user may need to perform gage factor and thermal output calibrations for his system to establish a stable reference gage output at 4 K before beginning tension tests. For this reason, strain gage calibrations may be more difficult than extensometer calibrations (see 6.9.3.3).

NOTE 11—Some gage manufacturers provide estimated values of the gage factors for the use of their products at low temperatures. Their estimates do not necessarily agree with published research; therefore calibration by controlled experimental determinations is preferred. Gage factors at temperatures as low as 4 K for some common materials have been published in a few studies. For example, findings for Ni-Cr alloy gages show that the gage factor increases nonlinearly by 2.5 or 5 % as the temperature is reduced from 295 to 4 K (21.9 to −269°C or 71.3 to −452.5°F) (**12-14**).

6.9.2.2 *Selection and Characteristics*—Select a satisfactory combination of gage active element, backing material, and bonding agent based on experience and manufacturer's recommendations.

NOTE 12—Not every type of strain gage is usable at cryogenic temperatures. A common choice for extreme cryogenic service is a Ni-Cr-Al-Fe alloy gage with a temperature-compensated active element (**8**). A closed-face, (encapsulated) gage is preferable to an open-face gage to minimize grid surface bubbling due to the strain gage excitation voltage, typically 2 V. The bubbles create a noisy strain signal. Typically the gage resistance is 120 or 350 Ω, and a low excitation voltage is used to reduce Joule heating at 4 K.

6.9.2.3 *Wiring*—Various circuits may be used for wiring strain gages.

NOTE 13—The choice depends on purpose and accuracy desired. One circuit that is satisfactory for tension tests according to this standard is the three-wire example, in Fig. 3. The three-wire circuit nullifies or eliminates thermally induced resistance changes in the leadwires if the wires R_{l1} and

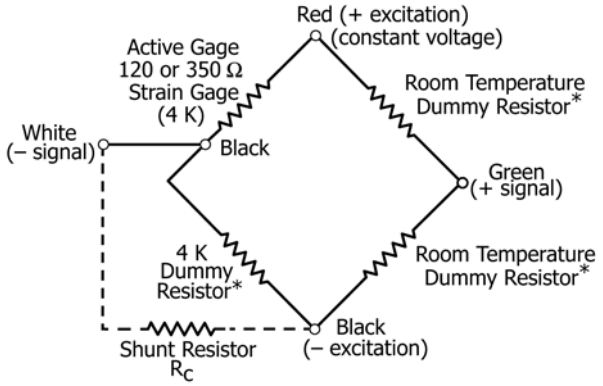


FIG. 3 A Method of Strain-Gage Wiring for Cryogenic Tests

$R_{/3}$ in Fig. 3 have the same resistance and experience the same temperature changes along their lengths. This circuit can be used with commercial strain indicators. First, balance the Wheatstone bridge at room temperature. Then, to compensate for any apparent strain induced on cooling the specimen and gage to the test temperature, rebalance the electrical signal after cooling before the force is applied and testing begins. Other circuits and instruments are possible, and some techniques offer higher accuracy than the illustrated example.

6.9.3 *Extensometers:*

6.9.3.1 *Types*—Reliable extensometers for use at 4 K may used.

NOTE 14—An example is the beam gage, which uses four strain-gage bonded in a full Wheatstone bridge. Extension within the specimen gage length is sensed by the detachable extensometer, which is clipped to retaining pins that are fixed to the specimen reduced section.

6.9.3.2 *Characteristics*—To measure the 0.2 % offset yield strength, one or more Class B-2 or better extensometers, as identified in Test Method E83, shall be used unless all parties agree to an alternate method (see 9.6.2). Each extensometer shall meet the Class B-2 requirements of Test Method E83 at 4 K. Whenever possible, mount the extensometer(s) directly to the specimen reduced section.

NOTE 15—It may be desirable to use several extensometers to minimize the effect of bending, especially for brittle specimens. One method for obtaining multi-planar strains is to fasten a pair of collars to the specimen reduced section, using sharpened radial thumbscrews. The collars are made with detents (located on the inboard faces) to accept spring-loaded extensometer. This arrangement also serves to define a fixed gage length within the specimen reduced cross section.

6.9.3.3 *Calibration*—Calibrate extensometers at room temperature and at 4 K. If the calibration is known and proved to be accurate, linear, and reproducible, then room-temperature checks may be performed before each series of tests to indirectly verify the 4 K calibration. However, direct calibration at 4 K shall be performed periodically, especially if damage is suspected or repairs have been made.

NOTE 16—For calibrations at 4 K, a device such as a micrometer with vertical extension tubes may be used with the extensometer(s) mounted at the lower end and immersed in liquid helium.

6.9.4 *Capacitance Extensometers*—Extensometers that use capacitance measurement to monitor strain may be used (**11**).

NOTE 17—The type with overlapping concentric cylinders has an extended strain range, an output that is linear with displacement, and an adjustable sensitivity. The type with parallel plates has high sensitivity, but its output is nonlinear, and it must be compensated for the hyperbolic dependence of capacitance on displacement.

7. Sampling

7.1 Remove samples for tension testing from the material in its final condition to ensure that the properties measured are representative of the product. Allow for any superficial effects introduced by the cutting operations.

7.2 Remove specimens from locations thought to be most representative of the stock material, realizing that data for specimens taken from selected locations of a structure or material may not be representative of the whole. The conventional locations should normally be used:

7.2.1 For products 40 mm (1.6 in.) or less in thickness or diameter, the location should be at the center.

7.2.2 For products over 40 mm (1.6 in.) in thickness or diameter, the location should be midway from the surface to the center.

7.3 Choose a specimen size and shape based on the requirements necessary to obtain representative samples of the material, and on the test machine's force capacity (see 6.2).

7.4 Using the notation in 4 of Test Method and Definitions A370, specify the orientation of the specimen's axis relative to the most predominant of either the grain flow or the principal working direction of the final form of the stock.

8. Test Specimens

8.1 *General:*

8.1.1 *Types and Specifications*—Any specimen configuration cited in Test Methods E8/E8M may be used. Specifications for dimensions, tolerances, and surface finish are stated in 6.1 through 6.17 of those standards.

8.1.2 *Size*—Specimens from sheet or wire products having relatively small cross-sectional areas can be tested within the force capacities of conventional apparatus. Specimens from thick plate or bar products, however, shall be machined to a reduced cross-sectional area so the force capacity of the machine is not exceeded. The specimen in this case should have the same configuration as the standard specimen but with an appropriately reduced cross section.

8.2 *Round Bar Specimens:*

8.2.1 *Standard 4 K Specimens*—To meet the force limitations of conventional test machines, the round bar specimens in 8.2.1.1 and 8.2.1.2 are defined as standard for 4 K tests. The required dimensions and tolerances for these specimens are given in Table 1. Threaded or shouldered ends are common for gripping these specimens, and the requirement of 6.4.1 can be met by precise machining.

NOTE 18—A 12.5-mm (0.5-in.) diameter round bar is the standard configuration for room-temperature tests according to Test Methods E8/E8M. Specimens of that size, however, require high test forces to fracture strong alloys at 4 K. For example, 210 kN (47 208 lbf) is required to test typical AISI 304LN steel at 4 K, whereas 100 kN (22 480 lbf) is the limit for most test machines.

8.2.1.1 *Standard, Small Metric Specimens*—These specimens have a 7-mm (0.275-in.) diameter and a G-to-D ratio of 5:1.

TABLE 1 Standard Specimens for Room-Temperature Tests and Recommended Proportionally Reduced, Standard Small Specimens for 4-K Tests[A]

(a) Metric Versions G/D ratio = 5 (dimensions, mm)	Standard Specimen	Standard Small Specimen
Nominal Diameter	12.5	7
G, gauge length	62.5 ± 0.1	35 ± 0.1
D, diameter	12.5 ± 0.1	7 ± 0.1
R, fillet radius	10	7
A, length of the reduced section	75	42
(b) U.S. Customary Versions G/D ratio = 4 (dimensions, in.)	**Standard Specimen**	**Standard Small Specimen**
Nominal Diameter	0.5	0.25
G, gauge length	2.000 ± 0.005	1.000 ± 0.005
D, diameter	0.500 ± 0.010	0.250 ± 0.005
R, fillet radius	0.375	0.1875
A, length of the reduced section	2.25	1.25

[A]See also the notes to the appropriate figure in Test Methods E8/E8M.

8.2.1.2 *Standard, Small U.S. Customary Specimens*—These specimens have a 6.25-mm (0.250-in.) diameter and a G-to-D ratio of 4:1.

8.2.2 *Alternatives*—If the 4 K standard specimens described above are inappropriate for some reason, other sizes may be selected following the guidelines of Test Methods E8/E8M. The proportions of such specimens should be similar to those of the standard specimens (see Fig. 4 of this test method and the applicable figures in Test Methods E8/E8M).

8.2.3 *Subsize Specimens*—Special care in fabrication and testing is required for specimens with diameters less than 6 mm (0.236 in.). As the specimen size is reduced, factors such as machining, surface finish, and alignment show increasing importance. Also, to ensure polycrystalline deformation, some experimenters insist on the need for a minimum of ten grains per cross section; if the number of grains per cross section is approximately ten or less, note this in the report.

9. Procedure

9.1 *Marking and Measuring the Test Specimen:*

9.1.1 *Gauge Length*—Gauge marks may be lightly punched, scribed, or inked at appropriate locations on the reduced section. This is the preferred method. After marking the gauge length, measure it to the nearest 0.05 mm (0.002 in.). The conventional gauge length is five times the diameter for metric specimens or four times the diameter for U.S. customary specimens. If another gauge length is used for elongation measurements, describe it in the report.

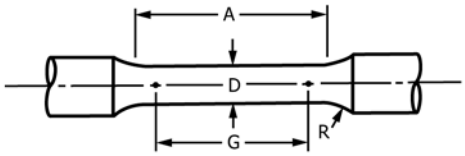


FIG. 4 Round Bar Specimen Configuration (see Table 1)

NOTE 19—For metals of low ductility, gauge marks punched or scribed on the reduced section may induce failure at those locations due to stress concentrations. To avoid this, coat the reduced section with layout ink, and mark the gauge length by rotating the specimen in a jig with knife edges scraping off the ink at the appropriate intervals. Otherwise, gauge marks may be placed on the specimen shoulders, or the overall length of the specimen may be used to determine elongations (see 9.6.5); in that case some error is introduced from measurement across section changes and the results should be qualified.

9.1.2 *Reduced Section and Overall Length*—Measure the length of the reduced section (A) and the adjusted length of the reduced section, if applicable, to the nearest 0.05 mm (0.002 in.). If overall specimen length (L) is used as a basis for determining elongations, measure the initial overall length of the specimen to the nearest 0.05 mm (0.002 in.).

9.1.3 *Cross Section*—Determine the cross-sectional area of the reduced section by measurements in accordance with the procedures of Test Methods E8/E8M.

9.2 *Specimen Installation*—Leaving sufficient slack for instrumentation wires so they will not be stretched or crimped during positioning of the Dewar or subsequent motions during testing, install the specimen in the tensile cryostat.

9.3 *Seating and Alignment*—If the gripping fixture involves loosely fitting components, such as spherically seated bearings, prevent friction or mismatch of the bearing surfaces at 4 K by first checking the seating and alignment at room temperature. During alignment, keep the applied tensile force below one-third of the proportional limit of the material being tested. Subsequently maintain a small but sufficient force to ensure that the alignment is retained during cool-down.

9.4 *Cooling Procedure*—Remove any condensate from the apparatus before cooling by drying it thoroughly with an air jet or heat gun. If a extensometer with a protective casing is used, position it so that cryogenic fluid can enter freely to surround the active elements to prevent the entrapment of gas bubbles and the associated extensometer noise.

NOTE 20—Ice can block cryogenic transfer lines or cause erratic force application behavior if it forms between various parts of the specimen, extensometer, and force train.

9.4.1 Begin testing only after the system has reached thermal equilibrium at 4 K. The specimen shall remain fully submerged at all times during the test. The liquid nitrogen level should be well above the top grip of the load train.

NOTE 21—The heat-transfer characteristics of gaseous helium are inferior to those of liquid helium; therefore it is imperative that the specimen remain submerged in liquid helium to minimize the influence of generated heat on the mechanical property measurements.

9.5 *Testing Speed:*

9.5.1 *Rate Control*—Since tensile property measurements in liquid helium are affected by testing speed, the test shall include a means of measuring and controlling the rate of crosshead motion. Calculate the nominal strain rate by dividing the crosshead rate by the length of the reduced section. Alternatively, a pacing or indicating device may be used to

monitor the strain rate, or an average strain rate may be determined by observing the time required to effect a known increment of strain.

NOTE 22—A nominal strain rate is specified, since the actual rate cannot be precisely controlled or maintained during discontinuous yielding.

9.5.2 *Rate Limit*—Any convenient crosshead speed may be used to reach an applied stress of one-half the yield strength; after that, the crosshead speed shall be chosen so that the nominal strain rate never exceeds 1×10^{-3} mm/mm/s.

NOTE 23—Higher rates can cause excessive specimen heating and therefore are not acceptable for basic mechanical property measurements of materials.

9.5.3 *Rate Selection*—Strain rates ranging from 10^{-5} to 10^{-3} mm/mm/s are generally recommended for tension tests at 4 K, but it may be desirable in some tests to use strain rates much lower than the 1×10^{-3} mm/mm/s maximum allowed by this test method.

NOTE 24—Some alloys are moderately sensitive to strain rate variations in this range. Some high strength austenitic steels show mild transitions in tensile properties at strain rates in the range 10^{-4} to 10^{-3} mm/mm/s, and other alloys with high ratios of strength to thermal conductivity (perhaps titanium alloys) may show similar trends (**15**).

9.5.4 *Rate Change*—The strain rate may be changed during a test.

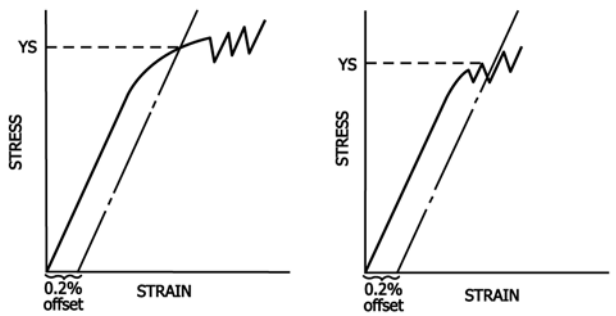
NOTE 25—For example, the strain required to initiate discontinuous yielding typically increases with decreasing strain rate. If the first serration occurs near 0.2 % plastic strain, plastic strain, reducing the speed of the test can postpone the first serration and prevent interference in the measurement of the yield strength (see Fig. 5). This can be accomplished by first using a relatively low strain rate to determine the yield strength, and then using a higher strain rate to complete the test.

9.6 *Measurement of Mechanical Properties:*

9.6.1 *Force-Extension Method*—To measure the yield strength, obtain a stress-strain diagram up to at least 0.2 % plastic strain.

9.6.1.1 Measure the yield strength by applying the 0.2 % offset method, following Test Methods E8/E8M. If the 0.2 % offset line intersects the curve at a decrease in force associated with discontinuous yielding, report the highest stress before that force decrease as the yield strength.

9.6.2 *Force-Time Method*—Yield strength measurements based on a 0.2 % offset procedure applied to force-versus-time (or force-versus-crosshead) curves at 4 K may be used for commercial test purposes with the agreement of all parties involved, but are not recommended. If this method or some other technique is used, state it clearly in the report.



A—Serrations after 0.2 % strain B—Serrations before 0.2 % strain

FIG. 5 Stress-Strain Diagram for Determination of Yield Strength by the Offset Method

NOTE 26—Force-time curves for tests at 4 K are typically nonlinear at the start and less regular than force-extension curves. The effective modulus of a thermally efficient force train may be low and depend on the liquid helium level. Also the time to achieve a steady state condition may be affected by changes in heat path efficiency through the increase of force upon nesting of pull rod interfaces. As a consequence, yield strength data from force-time curves may be less accurate than those of the recommended method. Checking any calculations using time-based deformation against system compliance calibrations at the same temperature can reveal if these issues are significant. In addition, comparing the total system deflection with total specimen strain using post-test gauge mark extension (with allowance for specimen elastic spring-back, especially if the material is relatively brittle) can reveal strain management issues.

9.6.3 *Discontinuous Yielding Stress*—Calculate the stress corresponding to the point of initiation of the first discontinuous-yielding event by dividing the maximum force sustained at the beginning of the first measurable serration by the cross-sectional area of the specimen.

9.6.4 *Tensile Strength*—Calculate the tensile strength by dividing the maximum force sustained by the specimen during the tension test by the original cross-sectional area of the specimen.

9.6.5 *Elongation*—If possible, calculate the percent elongation from the change in gauge length according to Test Methods E8/E8M. Otherwise, calculate the percent elongation from the initial (A_o) and final (A_f) values of the reduced section length: $100 \cdot (A_f - A_o)/A_o$, or from the initial ($L_o$) and final ($L_f$) values of the overall length, and the initial length (A_o) of reduced section: $100 \cdot (L_f - L_o)/A_o$. Report the method used.

9.6.6 *Reduction of Area*—Calculate the percent reduction of area according to Test Methods E8/E8M.

9.6.7 *Rounding Reported Test Data:*

9.6.7.1 Round the calculated numerical test results for yield and tensile strength, elongation, and reduction of area in accordance with procedures recommended in Section 7 of Test Methods E8/E8M.

9.6.7.2 For purposes of determining conformance with these specifications, round other observed values or calculated values “to the nearest unit” in the last right-hand significant digit used in expressing the limiting value, in accordance with the rounding method of Section 6 of Practice E29 Using Significant Digits in Test Data to Determine Conformance with Specifications.

9.6.8 *Replacement Specimens*—If necessary, discard any invalid data and test replacement specimens as per Test Methods E8/E8M.

10. Report

10.1 *General:*

10.1.1 *Material Characterization*—Describe the test material, including manufacturing, processing, and metallurgical information.

10.1.2 *Specimen Characterization*—Describe the specimen location and its orientation relative to the principal working directions or grain flow of the stock. Also report the specimen dimensions, including the cross-section dimensions, the fillet radius, the reduced section length, and the adjusted length of the reduced section (if used).

10.1.3 *Strain Rate*—Report the crosshead speed and nominal strain rate for the entire test. If the rate was changed during the test, report the effective nominal strain rates before and after the rate was changed. If direct strain measurements were made from the fractured specimen, report the calculated rates from these data.

10.1.4 *Mechanical Property Measurements*—Report the yield strength, the tensile strength, the method of offset yield strength measurement, and the method of extension measurement. Report the region of attachment and the span of extensometer(s), if used. Also report the discontinuous yielding stress and the strain rate at which it was measured, the tensile elongation and the method of its calculation, the gauge-length-to-diameter ratio for cylindrical specimens, and the reduction of area.

10.2 *Optional Data*—Optional data of importance or interest the that may be reported include measurements of Young's modulus (requires a Class A or Class B1 extensometer) at 4 K, the average grain size of the test material, the room temperature mechanical properties, if measured or known, and the compliance of the test machine including the tensile cryostat.

10.3 *Replicate Tests*—If replicate specimens are tested, report the number of tests, the average value of all mechanical property measurements, and a measure of the scatter of the data.

10.4 *Subsize Specimens*—If subsize specimens are tested, state any precautions taken with respect to specimen machining, surface condition, or alignment, and report the grain size of the test material.

10.5 *Anomalies*—Report any anomalies in material behavior, test records, mode of failure, and type and location of fracture.

11. Precision and Bias

11.1 *Precision:*

11.1.1 *Interlaboratory Test Program*—The precision information given in Table 2 comes from an interlaboratory study **(16)** (ILS) conducted during the development of Test Method E1450 and analyzed in ASTM Research Report RR:E28-1046.[4] Six participating laboratories followed the requirements of Test Method E1450 and conducted four to eight tensile tests of a stainless steel (SUS 316LN). They reported yield strength, tensile strength, elongation, and reduction of area. Based on the published report, the interlaboratory study followed the current procedure of Test Method E1450.

11.1.2 *Test Result*—Table 2 summarizes precision information for 0.2 % offset yield strength, tensile strength, and reduction of area. It does not contain data for repeatability or reproducibility of elongation, because the participating laboratories used specimens with different gauge lengths. Research Report RR:E28-1046 contains the computations for elongation, however. The analysis in Research Report RR:E28-1046 followed the structure of E691, but used a random effects model and maximum likelihood estimation to account for the unequal sample sizes between the the laboratories.

11.1.3 *Definitions and Standard Deviations*—The terms repeatability standard deviation, repeatability limit, reproducibility standard deviation, and reproducibility limit are used as specified in Practice E177.

11.1.3.1 The repeatability limit *(r)* is the value below which the absolute difference between two individual test results obtained under repeatability conditions may be expected to occur with a probability of approximately 0.95 (95 %). It is 2.8 (~1.96 $2^{1/2}$) times the repeatability standard deviation. This multiplier is independent of the size of the interlaboratory study.

11.1.3.2 The reproducibility limit *(R)* is the value below which the absolute difference between two individual test results obtained under reproducibility conditions may be expected to occur with a probability of approximately 0.95 (95 %). It is 2.8 (~1.96 $2^{1/2}$) times the reproducibility standard

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E28-1046. Contact ASTM Customer Service at service@astm.org.

TABLE 2 Precision Statistics

Parameter	Average of cell averages	Repeatability Standard Deviation		Reproducibility Standard deviation		Repeatability limit	Reproducibility limit
	$\bar{X}$ (See[A])	s_r (See[B])	$\frac{s_r}{\bar{X}}$ (See[C])	S_r (See[D])	$\frac{S_r}{\bar{X}}$ (See[E])	r (See[F])	R (See[G])
Yield strength (offset = 0.2 %), S_y (MPa)	1067	11	1.03 %	15	1.41 %	31	42
Tensile strength S_u (MPa)	1715	11	0.64 %	29	1.69 %	31	81
Reduction of area, %	49.7	4.9	9.86 %	6.6	13.28 %	13.7	18.5

[A] $\bar{X}$ is the average of the cell averages, that is, the grand mean for the test parameter.
[B] s_r is the repeatability standard deviation (within-laboratory precision) in MPa for strengths or % for reduction of area.
[C] $\frac{s_r}{\bar{X}}$ is the coefficient of variation in %.
[D] S_r is the reproducibility standard deviation (between-laboratory precision) in MPa for strengths or % for reduction of area.
[E] $\frac{S_r}{\bar{X}}$ is the coefficient of variation in %.
[F] r is the 95 % repeatability limit in MPa for strengths or % for reduction of area.
[G] R is the 95 % reproducibility limit in MPa for strengths or % for reduction of area.

deviation. This multiplier is independent of the size of the interlaboratory study (that is, of the number of laboratories participating.)

11.2 *Bias*—No accepted standard values for the tensile properties of materials exist. In the absence of any true value, no meaningful statement can be made concerning the bias of data.

12. Keywords

12.1 adiabatic heating; cryogenic properties (of materials); discontinuous yielding; liquid helium; low temperature tests; mechanical properties (of materials); tensile cryostats; tensile properties; tension test

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