



# Standard Test Method for Tension Testing of Structural Alloys in Liquid Helium<sup>1</sup>

This standard is issued under the fixed designation E 1450; 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 ( $\epsilon$ ) 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 cryostat is fully submerged in normal liquid helium (He I) and tested using crosshead displacement control at a nominal strain rate of  $10^{-3} \text{ s}^{-1}$  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 elastic modulus is treated in Test Method E 111.

NOTE 1—The boiling point of normal liquid helium (He I) at sea level is 4.2 K ( $-269^\circ\text{C}$  or  $-452.1^\circ\text{F}$  or  $7.6^\circ\text{R}$ ). It decreases with geographic elevation and is 4.0 K ( $-269.2^\circ\text{C}$  or  $-452.5^\circ\text{F}$  or  $7.2^\circ\text{R}$ ) 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:

A 370 Test Methods and Definitions for Mechanical Testing of Steel Products<sup>2</sup>

E 4 Practices for Force Verification of Testing Machines<sup>3</sup>

E 6 Terminology Relating to Methods of Mechanical Testing<sup>3</sup>

E 8 Test Methods for Tension Testing of Metallic Materials<sup>3</sup>

E 8M Test Methods for Tension Testing of Metallic Materials [Metric]<sup>3</sup>

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications<sup>4</sup>

E 83 Practice for Verification and Classification of Extensometer System<sup>3</sup>

E 111 Test Method for Young's Modulus, Tangent Modulus, and Chord Modulus<sup>3</sup>

E 1012 Practice for Verification of Specimen Alignment Under Tensile Loading<sup>3</sup>

## 3. Terminology

### 3.1 Definitions:

3.1.1 The definitions of terms relating to tension testing that appear in Terminology E 6 shall apply here. The definitions in this section also apply.

3.1.2 *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.

3.1.3 *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 *axial strain*—the average of the longitudinal strains measured at opposite or equally spaced surface locations on the sides of the longitudinal axis of symmetry of the specimen. The longitudinal strains are measured using two or more strain-sensing devices located at the mid-length of the reduced section.

3.1.5 *bending strain*—the difference between the strain at the surface of the specimen and the axial strain (the bending strain varies around the circumference and along the reduced section of the specimen).

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

3.1.7 *discontinuous yielding stress,  $\sigma_f$* —the peak stress at the initiation of the first measurable serration on the curve of stress-versus-strain.

3.1.7.1 *Discussion*—The parameter  $\sigma_f$  is a function of test variables and is not a material constant.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee E28 on Mechanical Testing and is the direct responsibility of Subcommittee E28.10 on Effect of Temperature on the Properties of Metals.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 01.03.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 03.01.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 14.02.

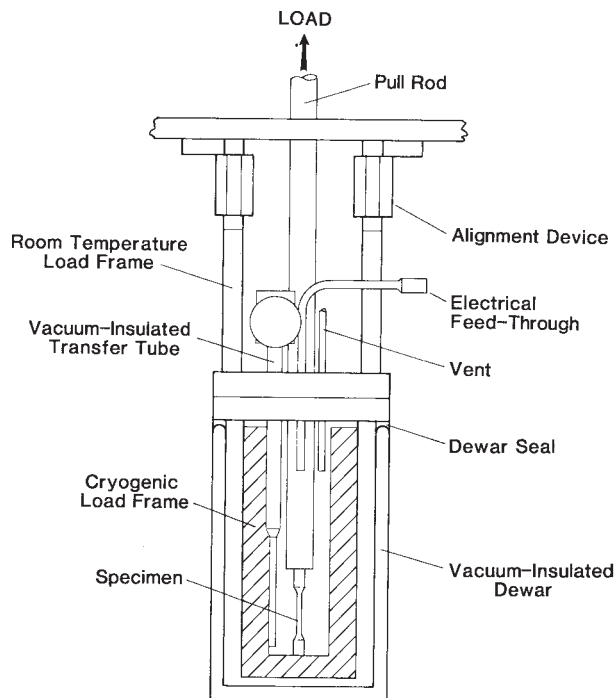


FIG. 1 Schematic Illustration of Typical Cryostat for Tension Testing at 4 K

3.1.8 *gage length*—the original distance between gage marks made on the specimen for determining elongation after fracture.

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

3.1.10 *maximum bending strain*—the largest value of bending strain in the reduced section of the specimen.

3.1.10.1 *Discussion*—Maximum bending strength is calculated from strains measured at two, three, or more circumferential positions, and at each of two different longitudinal positions.

3.1.11 *reduced section*—section in the central portion of the specimen, which has a cross section smaller than the gripped ends.

3.1.12 *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 alloys tested in liquid helium using displacement control are serrated (1).<sup>5</sup> 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 heat. 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 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 cryogenics 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 E 4 regarding verification of force accuracy. Know the test machine compliance (displacement per unit of applied force of the apparatus itself). 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 used to qualify the machine, as directed in 6.4.1 of this test method.

6.2 *System Design*—Typically, alloys in liquid helium exhibit double or triple their ambient strengths. For the same specimen geometry, higher forces must be applied to the cryostat, test specimen, force train members, and grips at cryogenic temperatures. Since many conventional test machines have a maximum force of 100 kN (22 480 lbf) or less,

<sup>5</sup> The boldface numbers in parentheses refer to the list of references at the end of this test method.

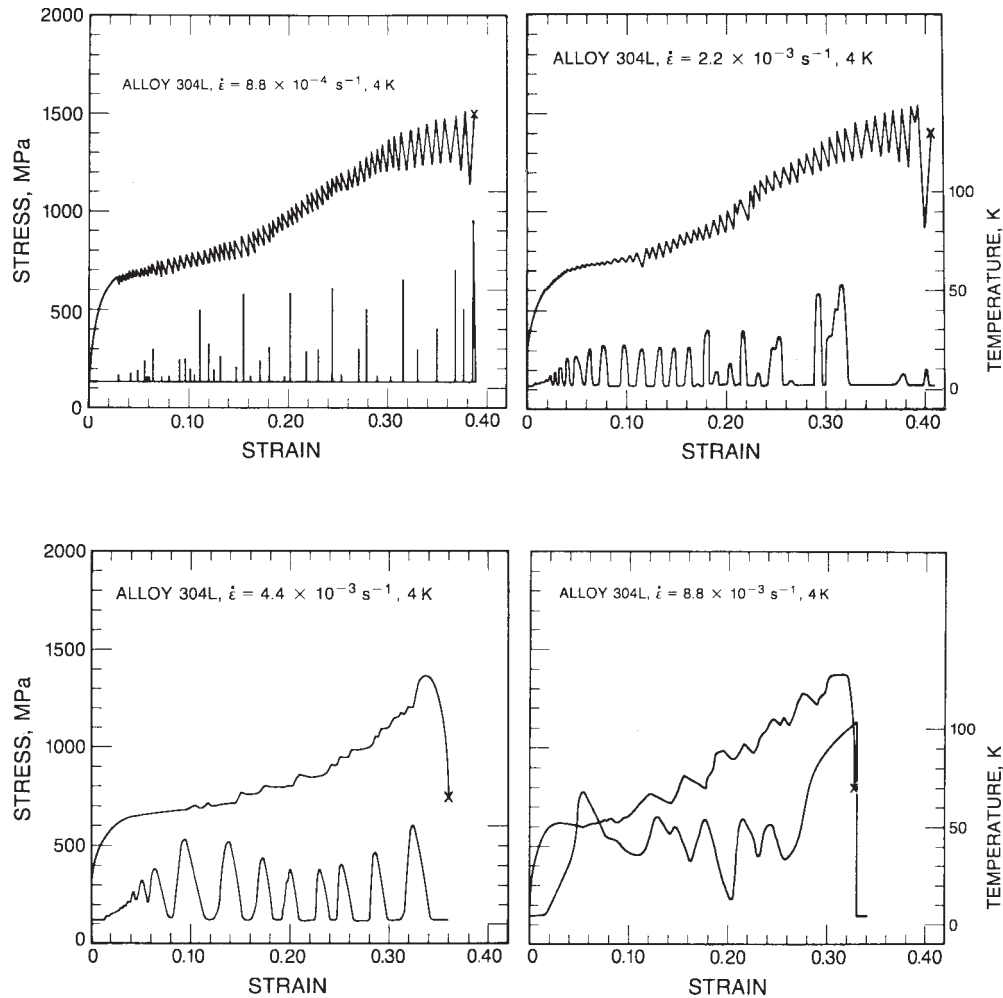


FIG. 2 Typical Engineering Stress-Strain Curves and Specimen Temperature Histories, at Four Different Nominal Strain Rates, for AISI 304L Stainless Steel Tested in Liquid Helium (4)

it is recommended that the apparatus be designed to accommodate one of the small specimens cited in 8.2.2 of this test method.

6.3 *Construction Materials*—Many construction materials, including the vast majority of ferritic steels, are brittle at 4 K. To prevent service failures, fabricate the grips and other force-train members using strong, tough, cryogenic alloys. 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 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 Proper system alignment is essential to avoid bending strains in the tension tests.

6.4.2 *Single-Specimen Apparatus*—For a conventional single-specimen cryostat, the machine and grips should be capable of applying force to a precisely machined calibration

specimen so that the maximum bending strain does not exceed 10 % of the axial strain. Reduce bending strain to an acceptable level by making proportional adjustments to a cryostat having alignment capability, or by using spacing shims to compensate an unadjustable fixture. Calculate the strain based on readings taken while the calibration specimen is subjected to a low force, as well as at the highest force for which the machine and force train are being qualified. Procedures for measuring specimen alignment are given in Practice E 1012.

NOTE 2—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.

6.4.3 *Multiple-Specimen Apparatus*—For this type of cryostat the alignment depends on the type of fixtures used. Measure and record the maximum bending strain.

6.4.4 Qualify the apparatus by making axiality measurements at room temperature and at 4 K. To perform axiality tests of the apparatus, the specimen form should be the same as that used during cryogenic tests, and the specimen concentricity should be as nearly perfect as possible. No plastic strain should

occur in the reduced section of the alignment specimen during application of force. In some cases this may necessitate the use of a relatively stiff, high-strength calibration specimen.

6.4.4.1 For cylindrical specimens, calculate the maximum bending strain defined in 3.1.10 from the strains measured at three circumferential positions, at each of two different longitudinal positions (if length permits). Measure the strains with three electrical-resistance strain gages, extensometers, or clip gages equally spaced around the reduced section of the specimen. The two longitudinal positions should be as far apart as possible, but not closer than one diameter to a fillet.

6.4.4.2 For specimens of square or rectangular cross section, measure the strain at the center of two parallel (opposite) faces, or in the case of thin cross sections, at the center of the two broad faces.

6.4.4.3 For conventional threaded or pinned grips, evaluate the effect of specimen bias as follows. Repeat the axiality measurements with the specimen rotated 180°, but with the grips and pull rods retained in their original positions. Then calculate the maximum bending strain and the strain at the specimen axis as the average of the two readings at the same position relative to the machine. If other grips or methods are used to evaluate the effect of specimen bias it should be described in the report.

6.4.5 *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. This section may be more appropriate under the strain gage section since it is referring to measurement of strain during the test and not alignment.

6.5 *Gripping Mechanisms*—The choice of gripping mechanism to be used is influenced by specimen type. The mechanisms described in Test Methods E 8 and E 8M are satisfactory at 4 K, but cryogenic materials must 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 which a given dimension must be measured.

#### 6.7 *Cryostats and Support Apparatus:*

6.7.1 *Cryostats*—A Dewar capable of retaining liquid helium is required. In general, cryostat force-application frames for existing test machines must be custom-built, but they may accommodate commercially available Dewars. The cryostat may employ adjustable force-columns to facilitate alignment. Several practical designs, including turret-disc designs for multiple-specimen testing with a single cooling, are discussed in Refs (6-10).

6.7.2 *Dewars*—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.

6.7.3 *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. Recycling or reliquefaction requires large investments in purification and support systems.

6.8 *Temperature Maintenance and Liquid-Level Indicators*—The intended test condition is ensured by maintaining a liquid helium environment. With the specimen completely immersed, a thermocouple to measure its temperature is not required for routine tests. Instead, a simple indicator or meter is required to 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 cryostat may be used to verify that the liquid level always remains above the specimen. Alternatively, the liquid level may be continuously monitored using a superconducting wire sensor of appropriate length positioned vertically inside the cryostat.

NOTE 3—One indication of the system nearing and reaching a steady state condition is the amount of condensation flare. As liquid helium is transferred into the cryostat, the flare becomes visible when boiled-off helium contacts room temperature air at the vent of the cryostat (Fig. 1). As cool-down proceeds, the flare decreases to a slowly issuing cloud due to less active boiling as the internal temperature of the cryostat reaches operating temperature.

#### 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). 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. These effects must be accounted for, or considerable error in strain measurements may be introduced.

6.9.2.2 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).

6.9.2.3 *Selection and Characteristics*—Not every type of strain gage is usable at cryogenic temperatures. Select a satisfactory combination of gage active element, backing material, and bonding agent based on experience and manufacturer’s recommendations. 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. 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. The full-scale operating range is typically 1 % at room temperature and 2 % at 4 K.

6.9.2.4 *Wiring*—Various circuits may be used for wiring strain gages. 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  $R_{11}$  and  $R_{13}$  in Fig. 3 have the same resistance and experience the same temperature changes along their lengths. This circuit may 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 may offer higher accuracy than the illustrated example.

NOTE 4—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).

6.9.3 *Clip-Gage Extensometers:*

6.9.3.1 *Types*—Reliable clip-gage extensometers for use at 4 K may be purchased or built. An example is the beam gage,

which uses four strain-gage films 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 E 83, may be used. Each extensometer must meet the sensitivity and accuracy requirements of Test Method E 83 and must be tested to ensure accuracy at 4 K. Whenever possible, mount the extensometer(s) directly to the specimen reduced section.

NOTE 5—It may be desirable to use several extensometers to detect the amount 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 clip gages. 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. For calibrations at 4 K, a device such as a micrometer with vertical extension tubes can be used with the extensometer(s) mounted at the lower end and immersed in liquid helium. 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 must be performed periodically, especially if damage is suspected or repairs have been made.

6.9.4 *Capacitance Extensometers*—Extensometers that use capacitance measurement to monitor strain may be used (11). 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 Take 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 Cut 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 A 370, 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.

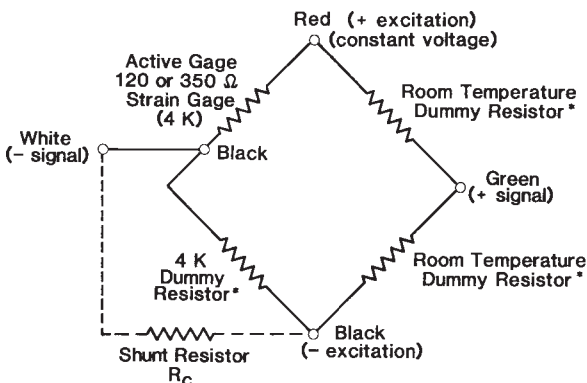


FIG. 3 A Method of Strain-Gage Wiring for Cryogenic Tests

8. Test Specimens

8.1 General:

8.1.1 *Types and Specifications*—Any specimen configuration cited in Test Methods E 8 or E 8M 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, must be machined to a reduced cross-sectional area so the force capacity of the machine is not exceeded. Preferably, 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 Room-Temperature Specimens*—A 12.5-mm (0.5-in.) diameter round bar is the standard configuration for room-temperature tests according to Test Methods E 8 and E 8M. 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.

8.2.2 *Standard 4 K Specimens*—To meet the force limitations of conventional test machines, the round bar specimens in 8.2.2.1 and 8.2.2.2 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.

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

8.2.2.2 *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.

8.2.3 *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 E 8 and E 8M. 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 E 8 or E 8M).

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

NOTE 6—For metals of low ductility, gage 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 gage length by rotating the specimen in a jig with knife edges scraping off the ink at the appropriate intervals. Otherwise, gage 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 E 8 and E 8M.

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 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.

TABLE 1 Standard Specimens for Room-Temperature Tests and Recommended Proportionally Reduced, Standard Small Specimens for 4-K Tests<sup>A</sup>

(a)	Metric Versions G/D ratio = 5 (dimensions, mm)	Standard Specimen	Standard Small Specimen
	Nominal Diameter	12.5	7
	G, gage length	62.5 ± 0.1	35 ± 0.1
	D, diameter	12.5 ± 0.1	7 ± 0.1
	R, fillet radius	10	7
	A, 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, gage 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, reduced section	2.25	1.25

<sup>A</sup>See also the notes to the appropriate figure in Test Methods E 8 and E 8M.

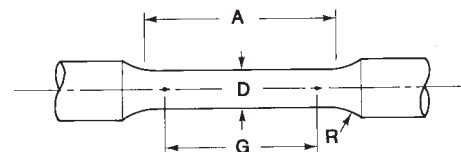


FIG. 4 Round Bar Specimen Configuration (see Table 1)

Subsequently maintain a small but sufficient force to ensure that the alignment is retained during cool-down.

**9.4 Cooling Procedure**—Ice can block cryogenic transfer lines or cause erratic force application behavior if it forms between various parts of the specimen, clip gage, and force train. To prevent icing, remove any condensate from the apparatus before cooling by drying it thoroughly with an air jet or heat gun. If a clip gage with a protective casing is used, position the gage so that cryogenic fluid can enter freely to surround the gage’s active elements to prevent the entrapment of gas bubbles and the associated clip gage noise.

**9.4.1** Next, position the Dewar and precool the apparatus by transferring liquid nitrogen into the cryostat. Monitor and maintain small force while cooling. After boiling subsides (an indication that thermal equilibrium is reached) remove all the liquid nitrogen from the cryostat, and transfer liquid helium into the cryostat until the specimen and grips are fully submerged. Testing may begin after the system has reached thermal equilibrium at 4 K. The specimen must remain fully submerged at all times during the test.

**NOTE 7**—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 must include a means of measuring and controlling the rate of crosshead motion. A nominal strain rate must be specified, since the actual rate cannot be precisely controlled or maintained in view of discontinuous yielding. The nominal strain rate is calculated 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.

**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 must be chosen so that the nominal strain rate never exceeds  $1 \times 10^{-3} \text{ s}^{-1}$ . Higher rates may 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  $10^{-3} \text{ s}^{-1}$  are generally recommended for tension tests at 4 K, but 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  $10^{-3} \text{ s}^{-1}$ , and other alloys with high ratios of strength to thermal conductivity (perhaps titanium alloys) may show similar trends (15). Consequently, it may be desirable in some tests to use strain rates much lower than the  $1 \times 10^{-3} \text{ s}^{-1}$  maximum allowed by this test method.

**9.5.4 Rate Change**—It may be desirable to change the strain rate during a test. 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, it may be possible to reduce the speed of the test to postpone

the first serration, and to prevent interference in the measurement of the yield strength (see Fig. 5). This may 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, a record of force-versus-extension must be obtained up to at least 0.2 % plastic strain. The use of a strain measurement device for autographic recording is recommended.

**9.6.1.1** Measure the yield strength by applying the 0.2 % offset method, following Test Methods E 8 or E 8M. If the 0.2 % offset line intersects the curve at a decrease in force associated with discontinuous yielding, then the highest stress before that force decrease is reported as the yield strength.

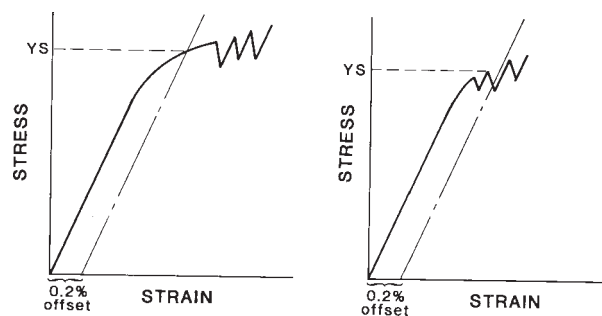
**9.6.2 Force-Time Method**—Yield strength measurements based on a 0.2 % offset procedure applied to force-versus-time (or force-versus-crosshead) curves at 4 K are generally not recommended, but may be used for commercial test purposes with the agreement of all parties involved. If this method or some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

**NOTE 8**—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 dependent 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. For this reason, check any calculations using time-based deformation against system stiffness calibrations at the same temperature. In addition, compare the total system deflection with total specimen strain using post-test gage mark extension (with allowance for specimen elastic spring-back, especially if the material is relatively brittle).

**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 gage length according to Test Methods



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**

E 8 or E 8M. 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:  $100 \cdot (L_f - L_o) / A_o$ .

9.6.6 *Reduction of Area*—Calculate the percent reduction of area according to Test Methods E 8 or E 8M.

9.6.7 *Rounding Reported Test Data*—Round off the calculated numerical test results in accordance with the procedures of Practice E 29 or other procedures recommended in Test Methods E 8 or E 8M.

9.6.8 *Replacement Specimens*—If necessary, discard any invalid data and test replacement specimens as per Test Methods E 8 or E 8M.

## 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 gage-length-to-diameter ratio for cylindrical specimens, and the reduction of area.

10.2 *Optional Data*—Report any optional data of importance or interest, such as measurements of Young’s modulus (requires a Class A or Class B1 extensometer) at 4 K, the average grain size of the test material, or the room temperature mechanical properties, if measured or known. Report the compliance of the test machine including the 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*—An overall measure of variability in tensile property measurements covered in this test method is being established by a series of interlaboratory tests.

11.2 *Bias*—There are no accepted standard values for the tensile properties of materials. 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); cryostats; discontinuous yielding; liquid helium; low temperature tests; mechanical properties (of materials); tensile properties; tension test

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