Designation: ~~E1450 – 09~~ 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} ~~s~~mm/mm/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~~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:*~~*Definitions of Terms Common to Mechanical Testing—*

3.1.1 The definitions of ~~terms relating to tension testing~~ mechanical testing terms that appear in the Terminology E6 ~~shall apply here. The definitions in this section also apply.~~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.

[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 ~~June 1, 2009~~Nov. 15, 2016. Published ~~August 2009~~February 2017. Originally approved in 1992. Last previous edition approved in ~~2003~~2009 as ~~E1450 – 03.~~E1450 – 09. DOI: ~~10.1520/E1450-09.~~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.

~~3.1.2 *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.~~

3.1.2 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 ~~*axial strain*—~~*discontinuous yielding, n*—~~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.~~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 *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.~~

3.1.5 *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.

3.1.6 ~~*gage length*—~~*gauge length, n*—the original ~~distance between gage marks made on the specimen for determining elongation after fracture.~~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*—

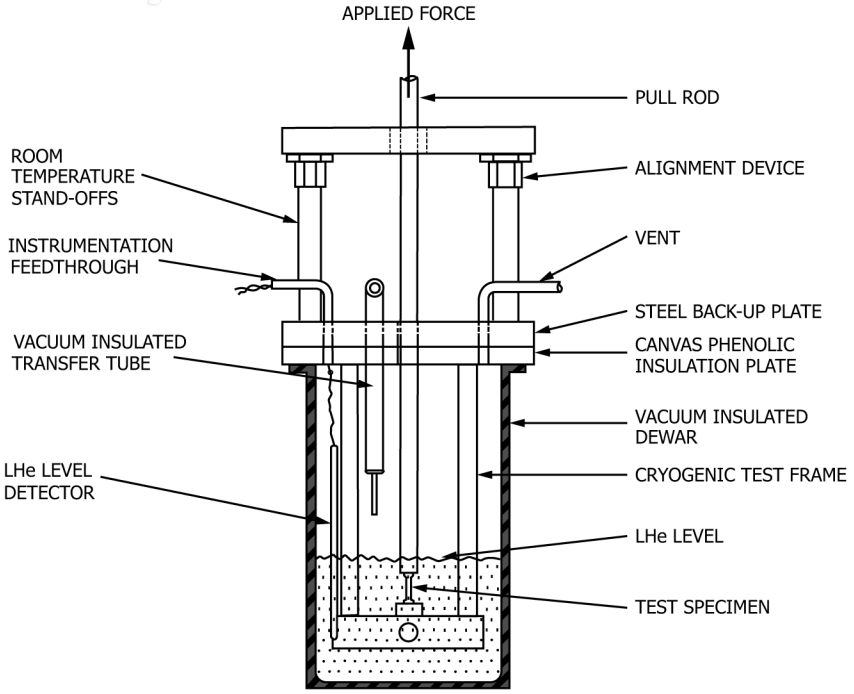

FIG. 1 Schematic Illustration of Typical Tensile Cryostat for Tension Testing at 4 K

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.

~~3.1.10 *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.~~

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

3.1.8.1 *Discussion*—

The cross section is uniform within prescribed tolerances.

~~3.1.12 *tensile cryostat*—a test apparatus for applying tensile forces to test specimens in cryogenic environments (Fig. 1).~~

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 ~~heat.~~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 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**).

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

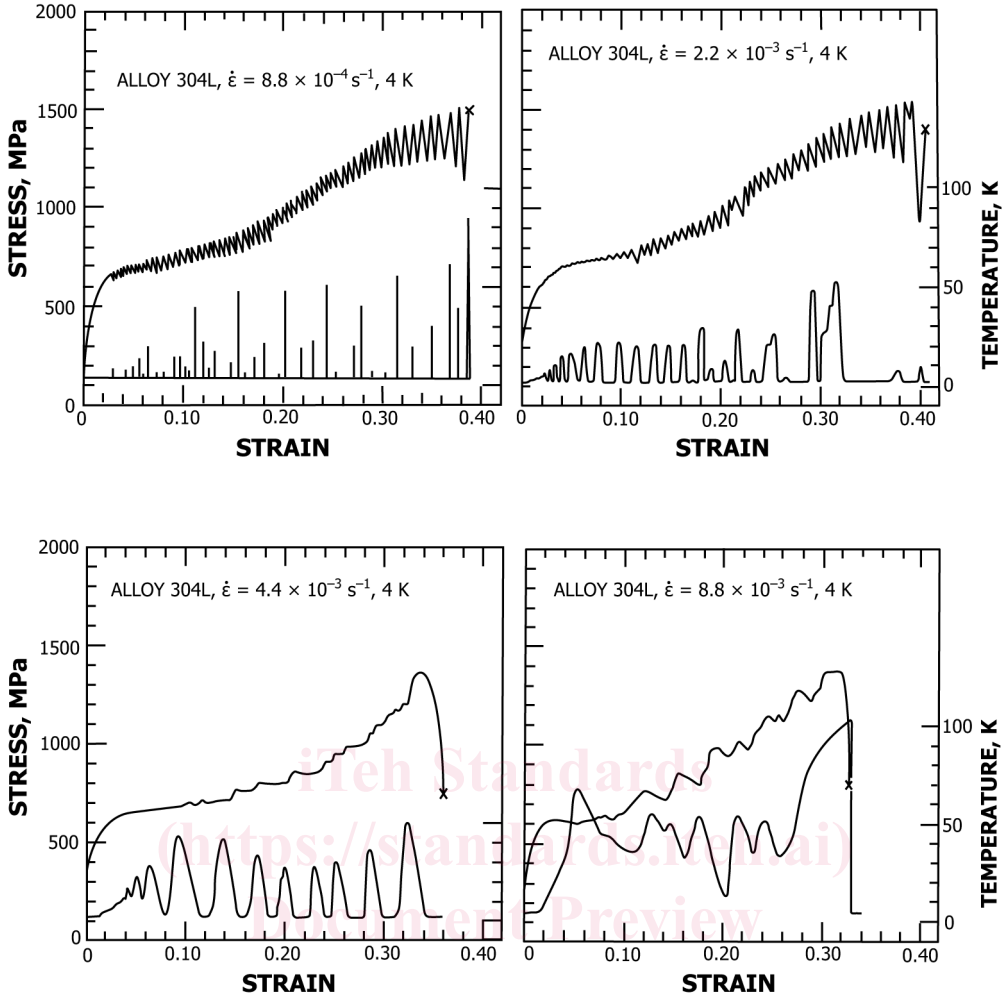

FIG. 2 Typical Engineering Stress-Strain Curves and Specimen Temperature Histories, at Four Different Nominal Strain Rates, for AISI 304L Stainless Steel Tested in Liquid Helium (4)

6. Apparatus

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

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. ~~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, it is recommended that the apparatus~~ The apparatus may be designed to accommodate one of the small specimens cited in ~~8.2.2~~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*—~~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.~~

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6.4 *Alignment:*

~~6.4.1 Proper system alignment is essential to avoid bending strains in the tension tests.~~

6.4.1 ~~*Single-Specimen Apparatus*—For a conventional single-specimen cryostat, the machine and grips should be capable of applying~~Single- and multiple-specimen systems shall meet Practice E1012 ~~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~~ Class 10 alignment at room temperature. ~~E1012.~~

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.

~~6.4.3 *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 E8/E8M are satisfactory at 4 K, but cryogenic materials ~~must~~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 ~~which a given dimension must~~ be measured.

6.7 *Tensile 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).~~

6.7.1 ~~*Dewars*—~~*Tensile Cryostats*—~~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~~ The ~~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.~~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**).

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

NOTE 7—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~~ Ensure that 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.~~ in liquid helium during the test.

NOTE 8—~~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 (~~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 ~~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.~~ 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**). ~~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.~~

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 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).~~

6.9.2.2 *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.~~

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