

Designation: C1337 - 10 (Reapproved 2015)

Standard Test Method for Creep and Creep Rupture of Continuous Fiber-Reinforced Advanced Ceramics Under Tensile Loading at Elevated Temperatures¹

This standard is issued under the fixed designation C1337; 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 covers the determination of the time-dependent deformation and time-to-rupture of continuous fiber-reinforced ceramic composites under constant tensile loading at elevated temperatures. This test method addresses, but is not restricted to, various suggested test specimen geometries. In addition, test specimen fabrication methods, allowable bending, temperature measurements, temperature control, data collection, and reporting procedures are addressed.
- 1.2 This test method is intended primarily for use with all advanced ceramic matrix composites with continuous fiber reinforcement: unidirectional (1-D), bidirectional (2-D), and tridirectional (3-D). In addition, this test method may also be used with glass matrix composites with 1-D, 2-D, and 3-D continuous fiber reinforcement. This test method does not address directly discontinuous fiber-reinforced, whisker-reinforced, or particulate-reinforced ceramics, although the test methods detailed here may be equally applicable to these composites.
 - 1.3 Values expressed in this test method are in accordance with the International System of Units (SI) and IEEE/ASTM SI 10
- 1.4 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. Hazard statements are noted in 7.1 and 7.2.

2. Referenced Documents

2.1 ASTM Standards:²

C1145 Terminology of Advanced Ceramics

C1275 Test Method for Monotonic Tensile Behavior of Continuous Fiber-Reinforced Advanced Ceramics with Solid Rectangular Cross-Section Test Specimens at Ambient Temperature

D3878 Terminology for Composite Materials

E4 Practices for Force Verification of Testing Machines

E6 Terminology Relating to Methods of Mechanical Testing

E83 Practice for Verification and Classification of Extensometer Systems

E139 Test Methods for Conducting Creep, Creep-Rupture, and Stress-Rupture Tests of Metallic Materials

E220 Test Method for Calibration of Thermocouples By Comparison Techniques

E230 Specification and Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples

E337 Test Method for Measuring Humidity with a Psychrometer (the Measurement of Wet- and Dry-Bulb Temperatures)

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

IEEE/ASTM SI 10 American National Standard for Use of the International System of Units (SI): The Modern Metric System

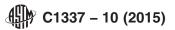
3. Terminology

3.1 Definitions:

¹ This test method is under the jurisdiction of ASTM Committee C28 on Advanced Ceramics and is the direct responsibility of Subcommittee C28.07 on Ceramic Matrix Composites.

Current edition approved $\frac{Dec.\ 1,\ 2010}{Duly\ 1,\ 2015}$. Published $\frac{January\ 2011}{September\ 2015}$. Originally approved in 1996. Last previous edition approved in $\frac{2005}{2010}$ as $\frac{C1337-96}{C1337-10.105}$. DOI: $\frac{10.1520/C1337-10.10520/C1337-10.$

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.



- 3.1.1 The definitions of terms relating to tensile testing appearing in Terminology E6 apply to the terms used in this test method. The definitions relating to advanced ceramics appearing in Terminology C1145 apply to the terms used in this test method. The definitions of terms relating to fiber reinforced composites appearing in Terminology D3878 apply to the terms used in this test method. Additional terms used in conjunction with this test method are defined in the following:
- 3.1.2 *continuous fiber-reinforced ceramic matrix composite* (CFCC)—ceramic matrix composite in which the reinforcing phase consists of a continuous fiber, continuous yarn, or a woven fabric.
- 3.1.3 fracture strength (F/L^2) —tensile stress that the material sustains at the instant of fracture. Fracture strength is calculated from the force at fracture during a tension test carried to rupture and the original cross-sectional area of the test specimen.

3.1.3.1 Discussion—

In some cases, the fracture strength may be identical to the tensile strength if the load at fracture is the maximum for the test. Factors such as load train compliance and fiber pull-out behavior may influence the fracture strength.

3.1.4 proportional limit stress—greatest stress which a material is capable of sustaining without any deviation from proportionality of stress to strain (Hooke's law).

3.1.4.1 Discussion—

Many experiments have shown that values observed for the proportional limit vary greatly with the sensitivity and accuracy of the testing equipment, eccentricity of loading, the scale to which the stress-strain diagram is plotted, and other factors. When determination of proportional limit is required, the procedure and sensitivity of the test equipment shall be specified.

3.1.5 *slow crack growth*—subcritical crack growth (extension) which may result from, but is not restricted to, such mechanisms as environmentally assisted stress corrosion or diffusive crack growth.

4. Significance and Use

- 4.1 This test method may be used for material development, material comparison, quality assurance, characterization, and design data generation.
- 4.2 Continuous fiber-reinforced ceramic matrix composites are candidate materials for structural applications requiring high degrees of wear and corrosion resistance and toughness at high temperatures.
- 4.3 Creep tests measure the time-dependent deformation of a material under constant load at a given temperature. Creep rupture tests provide a measure of the life of the material when subjected to constant mechanical loading at elevated temperatures. In selecting materials and designing parts for service at elevated temperatures, the type of test data used will depend on the criteria for load-carrying capability which best defines the service usefulness of the material.
- 4.4 Creep and creep rupture tests provide information on the time-dependent deformation and on the time-of-failure of materials subjected to uniaxial tensile stresses at elevated temperatures. Uniform stress states are required to effectively evaluate any nonlinear stress-strain behavior which may develop as the result of cumulative damage processes (for example, matrix cracking, matrix/fiber debonding, fiber fracture, delamination, etc.) which may be influenced by testing mode, testing rate, processing or alloying effects, environmental influences, or elevated temperatures. Some of these effects may be consequences of stress corrosion or subcritical (slow) crack growth. It is noted that ceramic materials typically creep more rapidly in tension than in compression. Therefore, creep data for design and life prediction should be obtained in both tension and compression.
- 4.5 The results of tensile creep and tensile creep rupture tests of specimens fabricated to standardized dimensions from a particular material or selected portions of a part, or both, may not totally represent the creep deformation and creep rupture properties of the entire, full-size end product or its in-service behavior in different environments or at various elevated temperatures.
- 4.6 For quality control purposes, results derived from standardized tensile test specimens may be considered indicative of the response of the material from which they were taken for given primary processing conditions and post-processing heat treatments.

5. Interferences

- 5.1 Test environment (vacuum, inert gas, ambient air, etc.) including moisture content (for example, relative humidity) may have an influence on the creep and creep rupture behavior of CFCCs. In particular, the behavior of materials susceptible to slow crack growth fracture and oxidation will be strongly influenced by test environment and test temperature. Testing can be conducted in environments representative of service conditions to evaluate material performance under these conditions.
- 5.2 Surface preparation of test specimens, although normally not considered a major concern with CFCCs, can introduce fabrication flaws which may have pronounced effects on the mechanical properties and behavior (for example, shape and level of the resulting stress-strain-time curve, etc.). Machining damage introduced during test specimen preparation can be either a random

interfering factor in the ultimate strength of pristine material (that is, increased frequency of surface-initiated fractures compared to volumeinitiated fractures) or an inherent part of the strength characteristics to be measured. Surface preparation can also lead to the introduction of residual stresses. Universal or standardized test methods of surface preparation do not exist. It should be understood that final machining steps may or may not negate machining damage introduced during the initial machining. Thus, test specimen fabrication history may play an important role in the measured time-to-failure or deformation, and shall be reported. In addition, the nature of fabrication used for certain composites (for example, chemical vapor infiltration or hot pressing) may require the testing of specimens in the as-processed condition (that is, it may not be possible to machine the test specimen faces without compromising the in-plane fiber architecture).

- 5.3 Bending in uniaxial tests does induce nonuniform stress distributions. Bending may be introduced from several sources including misaligned load trains, eccentric or misshaped specimens, and nonuniformly heated specimens or grips. In addition, if deformations or strains are measured at surfaces where maximum or minimum stresses occur, bending may introduce over or under measurement of strains depending on the location of the strain measuring device on the test specimen. Similarly, fracture from surface flaws may be accentuated or suppressed by the presence of the nonuniform stresses caused by bending.
- 5.4 Fractures that initiate outside the uniformly stressed gage section of a specimen may be due to factors such as stress concentrations or geometrical transitions, extraneous stresses introduced by gripping or thermal gradients, or strength limiting features in the microstructure of the test specimen. Such non-gage section fractures will normally constitute invalid tests. In addition, for face-loaded test specimen geometries, gripping pressure is a key variable in the initiation of fracture. Insufficient pressure can shear the outer plies in laminated CFCCs, while too much pressure can cause local crushing of the CFCC and lead to fracture in the vicinity of the grips.
- 5.5 The time-dependent stress redistribution that occurs at elevated temperatures among the CFCC constituents makes it necessary that the precise loading history of a creep test specimen be specified. This is of particular importance since the rate at which a creep load is initially applied can influence the subsequent creep behavior and damage modes. For example, whether matrix cracking would occur at the end of loading will depend on the magnitude of the loading rate, the test stress, the test temperature and the relative creep resistance of the matrix with respect to that of the fibers.^{3,4}
- 5.6 When CFCCs are mechanically unloaded either partially or totally after a creep test during which the test specimen accumulated time-dependent deformation, the specimen may exhibit creep recovery as manifested by a time-dependent reduction of strain. The rate of creep recovery is usually slower than the rate of creep deformation, and both creep and creep recovery are in most cases thermally activated processes, making them quite sensitive to temperature. Often it is desired to determine the retained strength of a CFCC after being subjected to creep for a prescribed period of time. Therefore, it is customary to unload the test specimen from the creep stress and then reload it monotonically until failure. Under these circumstances, the time elapsed between the end of the creep test and the conduction of the monotonic fast fracture test to determine the retained strength as well as the loading and unloading rates will influence the rate of internal stress redistribution among the phases and hence the CFCC strength.

https://standards.iteh.ai/catalog/standards/sist/0a353ecb-86b8-4f7a-b197-fb4be1cf8f37/astm-c1337-102015

6. Apparatus

6.1 Testing Machines—Machines used for tensile testing shall conform to the requirements of Practices E4. The forces used shall be accurate within ± 1 % at any force within the selected force range of the testing machine as defined in Practices E4.

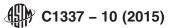
6.2 Gripping Devices:

6.2.1 General—Various types of gripping devices may be used to transmit the measured force applied by the testing machine to the test specimens. The brittle nature of the matrices of CFCCs requires that a uniform interface exists between the grip components and the gripped section of the specimen. Line or point contacts and nonuniform pressure can produce Hertzian-type stresses leading to crack initiation and fracture of the test specimen in the gripped section. Gripping devices can be classified generally as those employing active and those employing passive grip interfaces as discussed in the following sections. Grips located outside the heated zone surrounding the specimen may or may not employ cooling. Uncooled grips located outside the heated zone are termed warm grips and generally reduce the thermal gradient in the test specimen but at the expense of using high-temperature alloy grips and increased degradation of the grips due to exposure to the elevated-temperature environment. Cooled grips located outside the heated zone are termed cold grips and generally induce a steep thermal gradient along the length of the specimen.

Note 1—The expense of the cooling system for cold grips is balanced against maintaining alignment that remains consistent from test to test (stable grip temperature) and decreased degradation of the grips due to exposure to the elevated-temperature environment. When grip cooling is employed, provisions shall be provided to control the cooling medium to maximum fluctuations of 5 K (less than 1 K preferred) about a setpoint temperature over the course of the test to minimize thermally induced strain changes in the test specimen. In addition, opposing grip temperatures should be maintained at uniform and consistent temperatures not to exceed a difference ± 5 K (less than ± 1 K preferred) so as to avoid inducing unequal thermal gradients

³ Holmes, J. W., and Wu, X., "Elevated Temperature Creep Behavior of Continuous Fiber-reinforced Ceramics," *Elevated Temperature Mechanical Behavior of Ceramic Matrix Composites*, S. V. Nair and K. Jakus, eds., Butterworth-Heinneman, 1994.

⁴ Lara-Curzio, E., and Ferber, M. K., "Redistribution of Internal Stresses in Composite Materials During Creep," Ceram. Eng. Sci., 16, 5, 1995, pp. 791–800.



and subsequent nonuniaxial stresses in the specimen. Generally, the need for control of grip temperature fluctuations or differences may be indicated if test specimen gage section temperatures cannot be maintained within the limits prescribed in 9.2.2.

- 6.2.1.1 Active Grip Interfaces—Active grip interfaces require a continuous application of a mechanical, hydraulic, or pneumatic force to transmit the force to the test specimen. Generally, these types of grip interfaces cause a force to be applied normal to the surface of the gripped section of the test specimen. Transmission of the uniaxial load applied by the test machine is then accomplished by friction between the test specimen and the grip faces. Thus, important aspects of active grip interfaces are: (1) uniform contact between the gripped section of the test specimen and the grip faces, and (2) constant coefficient of friction over the grip/test specimen interface. In addition, note that fixed-displacement active grips set at ambient temperatures may introduce excessive gripping stresses due to thermal expansion of the test material when the test specimen is heated to the test temperature. Therefore, provisions shall be made to avoid such excessive stresses prior to the test by heating the test specimen while maintaining a constant force in the load train (for example, force control). Hydraulic grips are usually water cooled, and special provisions shall be made to ensure that these grips are continuously cooled since loss of cooling may result in rupture of the hydraulic lines and hydraulic chamber creating a potentially dangerous situation.
- (1) For flat test specimens, face-loaded grips, either by direct lateral pressure grip faces or by indirect wedge-type grip faces, act as the grip interface. Generally, close tolerances are required for the flatness and parallelism as well as for the wedge angle of the wedge grip faces. In addition, the thickness, flatness, and parallelism of the gripped section of the test specimen must be within similarly close tolerances to promote uniform contact at the test specimen/grip interface. Tolerances will vary depending on the exact test specimen configuration. For examples of tensile test specimen geometries, the user of this test method is referred to Test Method C1275.
- (1) For flat test specimens, face-loaded grips, either by direct lateral pressure grip faces or by indirect wedge-type grip faces, act as the grip interface. Generally, close tolerances are required for the flatness and parallelism as well as for the wedge angle of the wedge grip faces. In addition, the thickness, flatness, and parallelism of the gripped section of the test specimen must be within similarly close tolerances to promote uniform contact at the test specimen/grip interface. Tolerances will vary depending on the exact test specimen configuration. For examples of tensile test specimen geometries, the user of this test method is referred to Test Method C1275.
- (2) Sufficient lateral pressure must be applied to prevent slippage between the grip face and the test specimen. Grip surfaces that are scored or serrated with a pattern similar to that of a single-cut file have been found satisfactory. A fine serration appears to be the most satisfactory. The serrations shall be kept clean and well-defined but not overly sharp. The length and width of the grip faces shall be equal to or greater than the respective length and width of the gripped sections of the test specimen.
- (2) Sufficient lateral pressure must be applied to prevent slippage between the grip face and the test specimen. Grip surfaces that are scored or serrated with a pattern similar to that of a single-cut file have been found satisfactory. A fine serration appears to be the most satisfactory. The serrations shall be kept clean and well-defined but not overly sharp. The length and width of the grip faces shall be equal to or greater than the respective length and width of the gripped sections of the test specimen.
- 6.2.1.2 Passive Grip Interfaces—Passive grip interfaces transmit the force applied by the test machine to the test specimen through a direct mechanical link. Generally, these mechanical links transmit the test force to the test specimen by means of geometrical features of the test specimens such as shank shoulders or holes in the gripped head. Thus, the important aspect of passive grip interfaces is uniform contact between the gripped section of the test specimen and the grip faces.
- (1) For flat test specimens, passive grips may act either through edge-loading by means of grip interfaces at the shoulders of the test specimen shank or by combinations of face-loading and pin loading by means of pins at holes in the gripped head of the test specimen. Generally, close tolerances of linear and angular dimensions of shoulder and grip interfaces are required to promote uniform contact along the entire test specimen/grip interface as well as to provide for noneccentric loading. In addition, moderately close tolerances are required for center-line coincidence and diameters of the pins and hole. Examples of test specimen geometries adequate for passive grips are presented in Test Method C1275.
- (1) For flat test specimens, passive grips may act either through edge-loading by means of grip interfaces at the shoulders of the test specimen shank or by combinations of face-loading and pin loading by means of pins at holes in the gripped head of the test specimen. Generally, close tolerances of linear and angular dimensions of shoulder and grip interfaces are required to promote uniform contact along the entire test specimen/grip interface as well as to provide for noneccentric loading. In addition, moderately close tolerances are required for center-line coincidence and diameters of the pins and hole. Examples of test specimen geometries adequate for passive grips are presented in Test Method C1275.
- (2) When using edge-loaded test specimens, lateral centering of the test specimen within the grip attachments is accomplished by use of wedge-type inserts machined to fit within the grip cavity. Examples of successfully used edge-loaded test specimens are presented in Figs. 8 and Figs. 9 of Test Method C1275.
- (3) The pins in face/pin loaded grips (for such test specimens as those illustrated in Figs. 14 through 16 of Test Method C1275) are primarily for alignment purposes and force transmission. Secondary force transmission is through face-loading by means of mechanically actuated wedge grip faces. Proper tightening of the wedge grip faces against the test specimen to prevent slipping while avoiding compressive fracture of the test specimen gripped section must be determined for each material and test specimen type.

- (4) Note that passive grips employing single pins in each gripped section of the test specimen as the primary load transfer mechanism are not recommended. Relatively low interfacial shear strengths compared to longitudinal tensile strengths in CFCCs (particularly for 1-D reinforced materials loaded along the fiber direction) may promote non-gage section fractures along interfaces particularly at geometric transitions or at discontinuities such as holes.
- (2) When using edge-loaded test specimens, lateral centering of the test specimen within the grip attachments is accomplished by use of wedge-type inserts machined to fit within the grip cavity. Examples of successfully used edge-loaded test specimens are presented in Figs. 8 and Figs. 9 of Test Method C1275.
- (3) The pins in face/pin loaded grips (for such test specimens as those illustrated in Figs. 14 through 16 of Test Method C1275) are primarily for alignment purposes and force transmission. Secondary force transmission is through face-loading by means of mechanically actuated wedge grip faces. Proper tightening of the wedge grip faces against the test specimen to prevent slipping while avoiding compressive fracture of the test specimen gripped section must be determined for each material and test specimen type.
- (4) Note that passive grips employing single pins in each gripped section of the test specimen as the primary load transfer mechanism are not recommended. Relatively low interfacial shear strengths compared to longitudinal tensile strengths in CFCCs (particularly for 1-D reinforced materials loaded along the fiber direction) may promote non-gage section fractures along interfaces particularly at geometric transitions or at discontinuities such as holes.

6.3 Load Train Couplers:

- 6.3.1 General—Various types of devices (load train couplers) may be used to attach the active or passive grip interface assemblies to the testing machine. The load train couplers in conjunction with the type of gripping device play major roles in the alignment of the load train and thus subsequent bending imposed in the test specimen. Load train couplers can be classified generally as fixed and nonfixed as discussed in the following sections. Note that use of well-aligned fixed or self-aligning nonfixed couplers does not automatically guarantee low bending in the gage section of the tensile test specimen. Generally, well-aligned fixed or self-aligning nonfixed couplers provide for well-aligned load trains, but the type and operation of grip interfaces as well as the as-fabricated dimensions of the tensile test specimen can add significantly to the final bending imposed in the gage section of the test specimen.
- 6.3.1.1 Regardless of which type of coupler is used, alignment of the load train must be verified as a minimum at the beginning and end of a test series unless the conditions for verifying alignment as detailed in Section 11 of Test Method C1275 are otherwise met. A test series is interpreted to mean a discrete group of tests on individual test specimens conducted within a discrete period of time on a particular material configuration, test specimen geometry, test condition, or other uniquely definable qualifier. An additional verification of alignment is recommended, although not required, at the middle of the test series. Either a dummy or actual test specimen and the alignment verification procedures detailed in Section 11 of Test Method C1275 and Practice E1012 shall be used. Allowable bending requirements are discussed in 6.5. Tensile test specimens used for alignment verification shall be equipped with eight separate longitudinal strain gages to determine bending contributions from both eccentric and angular misalignment. Ideally the verification specimen shall be of identical material to that being tested. However, in the case of CFCCs the type of reinforcement or degree of residual porosity may complicate the consistent and accurate measurement of strain. Therefore, it is recommended that an alternate material (isotropic and homogeneous) with similar elastic modulus, elastic strain capability, and hardness to the test material be used. In addition, dummy specimens used for alignment verification shall have the same geometry and dimensions of the actual test specimens as well as similar mechanical properties as the test material to ensure similar axial and bending stiffness characteristics as the actual test specimen and material.
- 6.3.2 Fixed Load Train Couplers—Fixed couplers may incorporate devices which require either a one-time, pretest alignment adjustment of the load train which remains constant for all subsequent tests or an *in situ*, pretest alignment of the load train which is conducted separately for each test specimen and each test. Such devices usually employ angularity and concentricity adjusters to accommodate inherent load train misalignments. Regardless of which method is used, alignment verification must be performed as discussed in 6.3.1.1.
- 6.3.3 Nonfixed Load Train Couplers—Nonfixed couplers may incorporate devices which promote self-alignment of the load train during the movement of the cross-head or actuator. Generally such devices rely upon freely moving linkages to eliminate applied moments as the load train components are loaded. Knife edges, universal joints, hydraulic couplers, or air bearings are examples of such devices. Although nonfixed load train couplers are intended to be self-aligning and thus eliminate the need to evaluate the bending in the test specimen for each test, the operation of the couplers must be verified as discussed in 6.3.1.1.
- 6.3.3.1 Nonfixed load train couplers are useful in rapid test rate or constant load testing of CFCCs where the "graceful" fracture process is not as apparent. If the material exhibits graceful fracture the self-aligning feature of the nonfixed coupler will allow rotation of the gripped section of the specimen thus promoting a nonuniform stress in the remaining ligament of the gage section.

Note 2—Graceful fracture refers to the progressive process of matrix cracking and debonding and sliding of fibers that bridge those cracks and prevent the otherwise catastrophic mode of failure associated with brittle fracture.

- 6.4 Strain Measurement—Strain at elevated temperatures shall be determined by means of a suitable extensometer.
- 6.4.1 Extensometers used for tensile creep testing of CFCC test specimens shall satisfy Practice E83, Class B-1 requirements. Extensometers shall be calibrated periodically in accordance with Practice E83. For extensometers which mechanically contact the

test specimen, the contact shall not cause damage to the test specimen surface. In addition, extensometer contact probes must be chosen to be chemically compatible with the test material. In addition, the weight of the extensometer shall be supported so as not to introduce bending greater than that allowed in 6.5. Finally, the tips of the probes of contacting extensometers and the magnitude of the contact force shall be configured (for example, sharp knife edges or chisel tips) so as to minimize slippage.

- 6.5 Allowable Bending—Studies of the effects of bending on the tensile creep and tensile creep rupture behavior of CFCCs do not exist. Until such information is forthcoming for CFCCs, this test method adopts the recommendations for tensile testing of monolithic advanced ceramics. Therefore, the recommended maximum allowable percent for test specimens tested under this test method is 5 %. For verification of test specimen alignment, refer to Practice E1012.
- 6.6 *Heating Apparatus*—The apparatus for and method of heating the test specimens shall provide the temperature control necessary to satisfy the requirement of 9.2.
- 6.6.1 Heating can be by indirect electrical resistance (heating elements), indirect induction through a susceptor, or radiant lamp with the test specimen in ambient air at atmospheric pressure unless other environments are specifically applied and reported. Note that direct resistance heating is not recommended for heating CFCCs due to possible differences of the electrical resistances of the constituent materials which may produce nonuniform heating of the test specimen.
- 6.7 Temperature-Measuring Apparatus—The method of temperature measurement shall be sufficiently sensitive and reliable to ensure that the temperature of the test specimen is within the limits specified in 9.2.
- 6.7.1 Primary temperature measurement shall be made with thermocouples in conjunction with potentiometers, millivoltmeters, or electronic temperature controllers or readout units, or both. Such measurements are subject to two types of error. Thermocouple calibration and instrument measuring errors initially produce uncertainty as to the exact temperature. Secondly, both thermocouples and measuring instruments may be subject to variations over time. Common errors encountered in the use of thermocouples to measure temperatures include calibration error, drift in calibration due to contamination or deterioration with use, lead-wire error, error arising from method of attachment to the test specimen, direct radiation of heat to the bead, heat-conduction along thermocouple wires, etc.
- 6.7.2 Temperature measurements shall be made with thermocouples of known calibration. Representative thermocouples shall be calibrated from each lot of wires used for making noble-metal (for example, platinum (Pt) or rhodium (Rh)) thermocouples. Except for relatively low temperatures of exposure, noble-metal thermocouples are eventually subject to error upon reuse. Oxidized noble-metal thermocouples shall not be reused without clipping back to remove wire exposed to the hot zone, re-welding, and annealing. Any reuse of noble-metal thermocouples after relatively low-temperature use without this precaution shall be accompanied by re-calibration data demonstrating that calibration was not unduly affected by the conditions of exposure.
- 6.7.3 Measurement of the drift in calibration of thermocouples during use is difficult. When drift is a problem during tests, a method shall be devised to check the readings of the thermocouples monitoring the test specimen temperature during the test. For reliable calibration of thermocouples after use, the temperature gradient of the test furnace must be reproduced during the re-calibration.
- 6.7.4 Temperature measuring, controlling, and recording instruments shall be calibrated against a secondary standard, such as precision potentiometer, optical pyrometer, or black-body thyristor. Lead-wire error shall be checked with the lead wires in place as they normally are used. For thermocouple calibration procedures, refer to Test Method E220 and Specification E230.
- 6.8 Data Acquisition—At the minimum, gage section elongation or strain versus time shall be obtained. Either analog chart recorders or digital data acquisition systems can be used for this purpose, although a digital record is recommended for ease of later data analysis. Ideally, an analog chart recorder or plotter should be used in conjunction with the digital data acquisition system to provide an immediate record of the test as a supplement to the digital record. Recording devices shall be accurate within ± 1 % of the selected range for the testing system including readout unit, as specified in Practices E4.
- 6.8.1 Cross-head displacement of the test machine may also be recorded but shall not be used to define displacement or strain in the gage section especially when self-aligning couplers are used in the load train.
- 6.8.2 Temperature shall be recorded at the initiation and completion of the actual test. However, temperature can also be recorded parallel to the strain record in addition to temperature recordings at the start of the heating of the furnace (including ramp-up to test temperature) and ending at the completion of the test.
- 6.9 *Dimension-Measuring Devices*—Micrometers and other devices used for measuring linear dimensions shall be accurate and precise to at least one half of the smallest unit to which the individual dimension is required to be measured. For the purposes of this test method, cross-sectional dimensions shall be measured to within 0.02 mm requiring dimension measuring devices with accuracies of 0.01 mm.

7. Hazard Statements

7.1 During the conduct of this test method, the possibility of flying fragments of broken test material may be high. The brittle nature of advanced ceramics and the release of strain energy contribute to the potential release of uncontrolled fragments upon fracture. Means for containment and retention of these fragments for later fractographic reconstruction and analysis is highly recommended.