Designation: C 1337 – 96 (Reapproved 2005)

Standard Test Method for Creep and Creep Rupture of Continuous Fiber-Reinforced Ceramic Composites under Tensile Loading at Elevated Temperatures[1]

This standard is issued under the fixed designation C 1337; 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, 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]

C 1145 Terminology on Advanced Ceramics

C 1275 Test Method for Monotonic Tensile Behavior of Continuous Fiber-Reinforced Advanced Ceramics with Solid Rectangular Cross-Section Test Specimens at Ambient Temperature

D 3878 Terminology for Composite Materials

E 4 Practices for Force Verification of Testing Machines

E 6 Terminology Relating to Methods of Mechanical Testing

E 83 Practice for Verification and Classification of Extensometer System

E 139 Practice for Conducting Creep, Creep Rupture, and Stress Rupture Tests of Metallic Materials

E 220 Test Method for Calibration of Thermocouples By Comparison Techniques

E 230 Specification for Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples

E 337 Test Method for Measuring Humidity with a Psychrometer (The Measurement of Wet- and Dry-Bulb Temperatures)

E 1012 Practice for Verification of Specimen Alignment under Tensile Loading

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*—The definitions of terms relating to tensile testing appearing in Terminology E 6 apply to the terms used in this test method. The definitions relating to advanced ceramics appearing in Terminology C 1145 apply to the terms used in this test method. The definitions of terms relating to fiber reinforced composites appearing in Terminology D 3878 apply to the terms used in this test method. Additional terms used in conjunction with this test method are defined in the following:

3.1.1 *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.

[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.

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[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 *fracture strength*—tensile stress which the material sustains at the instant of fracture. Fracture strength is calculated from the load at fracture during a tension test carried to rupture and the original cross-sectional area of the specimen.

3.1.2.1 *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.

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

3.1.3.1 *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.

3.1.4 *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 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, 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 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 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 specimen. Such non-gage section fractures will normally constitute invalid tests. In addition, for face-loaded 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 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 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 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.

6. Apparatus

6.1 *Testing Machines*—Machines used for tensile testing shall conform to the requirements of Practices E 4. The loads used shall be accurate within ±1 % at any load within the selected load range of the testing machine as defined in Practices E 4.

6.2 *Gripping Devices*:

6.2.1 *General*—Various types of gripping devices may be used to transmit the measured load 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 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 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 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 and subsequent nonuniaxial stresses in the specimen. Generally, the need for control of grip temperature fluctuations or differences may be indicated if 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 load to the test specimen. Generally, these types of grip interfaces cause a load to be applied normal to the surface of the gripped section of the specimen. Transmission of the uniaxial load applied by the test machine is then accomplished by friction between the specimen and the grip faces. Thus, important aspects of active grip interfaces are: (*1*) uniform contact between the gripped section of the specimen and the grip faces, and (*2*) constant coefficient of friction over the grip/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 specimen is heated to the test temperature. Therefore, provisions shall be made to avoid such excessive stresses prior to the test by heating the specimen while maintaining a constant force in the load train (for example, load 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 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 specimen must be within similarly close tolerances to promote uniform contact at the specimen/grip interface. Tolerances will vary depending on the exact specimen configuration. For examples of tensile specimen geometries, the user of this test method is referred to Test Method C 1275.

(*2*) Sufficient lateral pressure must be applied to prevent slippage between the grip face and the 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

[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.

width of the grip faces shall be equal to or greater than the respective length and width of the gripped sections of the specimen.

6.2.1.2 *Passive Grip Interfaces*—Passive grip interfaces transmit the load applied by the test machine to the test specimen through a direct mechanical link. Generally, these mechanical links transmit the test loads to the specimen by means of geometrical features of the 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 specimen and the grip faces.

(*1*) For flat specimens, passive grips may act either through edge-loading by means of grip interfaces at the shoulders of the specimen shank or by combinations of face-loading and pin loading by means of pins at holes in the gripped specimen head. Generally, close tolerances of linear and angular dimensions of shoulder and grip interfaces are required to promote uniform contact along the entire 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 specimen geometries adequate for passive grips are presented in Test Method C 1275.

(*2*) When using edge-loaded specimens, lateral centering of the 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 specimens are presented in Figs. 8 and Figs. 9 of Test Method C 1275.

(*3*) The pins in face/pin loaded grips (for such specimens as those illustrated in Figs. 14 through 16 of Test Method C 1275) are primarily for alignment purposes and load transmission. Secondary load transmission is through face-loading by means of mechanically actuated wedge grip faces. Proper tightening of the wedge grip faces against the specimen to prevent slipping while avoiding compressive fracture of the specimen gripped section must be determined for each material and specimen type.

(*4*) Note that passive grips employing single pins in each gripped section of the 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 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 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 specimen can add significantly to the final bending imposed in the gage section of the 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 C 1275 are otherwise met. A test series is interpreted to mean a discrete group of tests on individual 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 C 1275 and Practice E 1012 shall be used. Allowable bending requirements are discussed in 6.5. Tensile 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 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 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 CFCCs specimens shall satisfy Practice E 83, Class B-1 requirements. Extensometers shall be calibrated periodically in accordance with Practice E 83. For extensometers which mechanically contact the specimen, the contact shall not cause damage to the 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 specimens tested under this test method is 5 %. For verification of specimen alignment, refer to Practice E 1012.

6.6 *Heating Apparatus*—The apparatus for and method of heating the 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 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 specimen.

6.7 *Temperature-Measuring Apparatus*— The method of temperature measurement shall be sufficiently sensitive and reliable to ensure that the temperature of the 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 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 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 blackbody 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 E 220 and Specification E 230.

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 E 4.

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.

7.2 Exposed fibers at the edges of CFCC specimens present a hazard due to the sharpness and brittleness of the ceramic fiber. All persons required to handle these materials shall be well-informed of such conditions and the proper handling techniques.

8. Test Specimens

8.1 *Test Specimen Geometry*:

8.1.1 *General*—The geometry of tensile creep test specimens is dependent on the ultimate use of the tensile creep data. For example, if the tensile creep of an as-fabricated component is required, the dimensions of the resulting tensile specimen may reflect the thickness, width, and length restrictions of the component. If it is desired to evaluate the effects of interactions of various constituent materials for a particular CFCC manufactured by means of a particular processing route then the size of the specimen and resulting gage section will reflect the desired volume to be sampled. In addition, grip interfaces and load train couplers as discussed in Section 6 will influence the final design of the specimen geometry.

8.1.1.1 The following sections discuss the more common and, thus, proven specimen geometries, although any geometry is acceptable if it meets the gripping, fracture location, temperature profile, and bending requirements of this test method. Deviations from the recommended geometries may be necessary depending upon the particular CFCC being evaluated. Stress analyses of untried specimens shall be conducted to ensure that stress concentrations which can lead to undesired fractures outside the gage sections do not exist. It should be noted that contoured specimens by their nature contain inherent stress concentrations due to geometric transitions. Stress analyses can indicate the magnitude of such stress concentrations while revealing the success of producing a uniform tensile stress state in the gage section of the specimen. Additionally, the success of an elevated-temperature creep test will depend on the type of heating system, extent of specimen heating, and specimen geometry since these factors are all interrelated. For example, thermal gradients may introduce additional stress gradients in specimens which may already exhibit stress gradients at ambient temperatures due to geometric transitions. Therefore, untried test configurations should be simultaneously analyzed for both loading induced stress gradients and thermally induced temperature gradients to ascertain any adverse interactions.

8.1.1.2 Generally, specimens with contoured gage sections (transition radii of >50 mm) are preferred to promote the tensile stresses with the greatest values in the uniformly stressed gage section while minimizing the stress concentration due to the geometrical transition of the radius. However, in certain instances, (for example, 1-D CFCCs tested along the direction of the fibers) low interfacial shear strength relative to the tensile strength in the fiber direction will cause splitting of the specimen initiating at the transition region between the gage section and the gripped section of the specimen with the split propagating along the fiber direction leading to fracture of the specimen. In these cases, straight-sided specimens may be required for determining the tensile creep and creep rupture behavior of the CFCC. Figure 7 in Test Method C 1275 shows an example of a straight-sided specimen. In other instances, a particular fiber weave or processing route will preclude fabrication of specimens with reduced gage sections, thus requiring implementation of straight-sided specimens. Straight-sided specimens may be gripped by any of the methods discussed herein, although active gripping systems are recommended for minimizing non-gage section fractures.

8.1.2 *Edge-Loaded Flat Tensile Specimens*— This type of geometry has been successfully employed for the evaluation of 2-D and 3-D CFCCs. Of particular concern with this geometry is the proper and consistent angle of the edge-loaded shank. However, the preparation of this type of specimen with the stringent tolerances required is routine with numerical-controlled machines. Furthermore, this specimen is ideal when using "warm" or "hot" grips to minimize thermal gradients along the length of the specimen. Figures 8 and Figures 9 in Test Method C 1275 show examples of contoured edge-loaded specimens.

8.1.3 *Face-Loaded Flat Tensile Specimens*— This configuration exploits the friction at the specimen/grip interface to transmit the uniaxial load applied by the test machine. Important tolerances for the face-loaded geometry include parallelism and flatness of faces, all of which will vary depending on the exact configuration as shown in the appropriate specimen drawings.

8.1.3.1 For face-loaded specimens, especially for straight-sided (for example, noncontoured) specimens, end tabs may be required to provide a compliant layer for gripping. For CFCCs, fiberglass reinforced epoxy, PMR, and carbon fiber-reinforced resins, tab materials have been used successfully. However, metallic tabs (for example, aluminum alloys) may be satisfactory (or desirable for elevated temperature use) as long as the tabs are strain compatible (that is, having an elastic modulus of magnitude comparable to the bulk elastic modulus of the CFCC) with the CFCC material being tested. Each beveled tab (bevel angle $\leq 15°$) shall be a minimum of 30 mm long, the same width of the specimen, and have the total thickness of the tabs on the order of the thickness of the test specimen. Any high-elongation (tough) adhesive system may be used with the length of the tabs determined by the shear strength of the adhesive, size of the specimen, and estimated strength of the composite. In any case, a significant fraction (>10 to 20 %) of fractures within one specimen width of the tab shall be cause to reexamine the tab materials and configuration, gripping method, and adhesive, and to make necessary adjustments to promote fracture within the gage section. Note that care should be taken to ensure that both the adhesive and tab material are capable of use at the temperature which might occur in the grip region. Figure 13 in Test Method C 1275 shows an example of a bevelled tab.

8.1.4 *Pin/Face-Loaded Flat Tensile Specimens*—These specimens employ combinations of pin and face loading to transmit the uniaxial load of the test machine to the specimen. Close tolerances of hole/pin diameters and center lines are required to ensure proper specimen alignment in the grips and

transmission of the loads, since the face-loaded part of the geometry provides a secondary load transmission mechanism in these specimens. Important tolerances for the face-loaded part of the geometry include parallelism and flatness of faces, both of which will vary depending on the exact configuration as shown in the appropriate specimen drawings. Thus, the pin/face-loaded geometry may require somewhat intensive fabrication procedures. Figures 14 through 16 in Test Method C 1275 show examples of contoured, pin/face-loaded specimens.

8.1.4.1 Note that specimens requiring single pins in each gripped section of the 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.

8.2 *Specimen Preparation*:

8.2.1 Depending upon the intended application of the tensile creep data, use one of the following specimen preparation procedures. Regardless of the preparation procedure used, sufficient details regarding the procedure must be reported to allow replication.

8.2.2 *As-Fabricated*—The tensile specimen should simulate the surface/edge conditions and processing route of an application where no machining is used, for example, as-cast, sintered, or injection-molded part. No additional machining specifications are relevant. As-processed specimens might possess rough surface textures and nonparallel edges and as such may cause excessive misalignment or proneness to non-gage section fractures, or both.

8.2.3 *Application-Matched Machining*— The tensile specimen should have as close to the same surface/edge preparation as that given to the component. Unless the process is proprietary, the report shall be specific about the stages of material removal, wheel grits, wheel bonding, amount of material removed per pass, and type of coolant used.

8.2.4 *Customary Practices*—In instances where a customary machining procedure has been developed that is completely satisfactory for a class of materials (that is, it induces no unwanted surface/subsurface damage or residual stresses), this procedure shall be used.

8.2.5 *Standard Procedure*—In instances where 8.2.2 through 8.2.4 are not appropriate, 8.2.5 shall apply. Studies to evaluate the machinability of CFCCs have not been completed. Therefore, the standard procedure of 8.2.5 can be viewed as starting-point guidelines, and a more stringent procedure may be necessary.

8.2.5.1 All grinding or cutting shall be done with ample supply of appropriate filtered coolant to keep the workpiece and grinding wheel constantly flooded and particles flushed. Grinding can be done in at least two stages, ranging from coarse to fine rate of material removal. All cutting can be done in one stage appropriate for the depth of cut.

8.2.5.2 Stock removal rate shall be on the order of 0.03 mm per pass using diamond tools that have between 320 and 600 grit. Remove equal stock from each face where applicable.

8.3 *Handling Precaution*—Care should be exercised in storage and handling of finished specimens to avoid the introduction of random and severe flaws. In addition, attention shall be given to pre-test storage of specimens in controlled environments or desiccators to avoid unquantifiable environmental degradation of specimens prior to testing.

8.4 *Specimen Sampling and Number*—Samples of the material to provide test specimens must be taken from such locations so as to be representative of the billet or lot from which it is taken. Although each testing scenario will vary, a typical designed experiment may include creep tests at stresses below, about, and above the monotonic matrix-cracking stress level and at least for one stress level, tests across a range of four temperatures. It is recommended that at least 20 % of the tests in the designed experiment be replicated (duplicated or triplicated) to determine levels of repeatability.

9. Procedure

9.1 *Specimen Dimensions*—Determine the thickness and width of the gage section of each specimen to within 0.02 mm. Make measurements on at least three different cross-sectional planes in the gage section. To avoid damage in the gage section area, these measurements shall be made either optically (for example, an optical comparator) or mechanically using a flat, anvil-type micrometer. In either case, the resolution of the instrument shall be as specified in 6.9. Exercise caution to prevent damage to the specimen gage section. Ball-tipped or sharp-anvil micrometers are not recommended because localized cracking can be induced. The measured dimensions and locations of the measurements shall be recorded and reported for use in the calculation of the tensile stress. The average of multiple measurements shall be used in the stress calculations.

9.1.1 Alternatively, to avoid damage to the gage section, make post-fracture measurements of the gage section dimensions using the procedures described in 9.1. Note that in some cases, the fracture process can severely fragment the gage section in the immediate vicinity of the fracture thus making post-fracture measurements of dimensions difficult. In these cases, follow the procedures outlined in 9.1 for pretest measurements to assure reliable measurements.

9.1.2 Conduct periodic, if not 100 %, inspection/measurements of all specimens and specimen dimensions to assure compliance with the drawing specifications. Generally, high-resolution optical methods (for example, an optical comparator) or high-resolution digital point contact methods (for example, coordinate measurement machine) are satisfactory as long as the equipment meets the specifications prescribed in 6.9. Note that the frequency of gage section fractures and bending in the gage section are dependent on proper overall specimen dimensions within the required tolerances.

9.1.3 In some cases it is desirable, but not required, to measure surface finish to quantify surface condition. Such methods as contacting profilometry can be used to determine surface roughness perpendicular to the tensile axis. When quantified, surface roughness shall be reported.

9.1.4 *Strain Measurement*:

9.1.4.1 *Optical Method*—Two or four flags of dimensions suitable for the gage width and thickness are attached to the specimen gage length. The flags are made by cutting an angled

slot at one end of a rectangular piece of a suitable high-temperature material (preferably the same material as the test specimen or sintered SiC if the test material is not available) with a gap the size of the specimen cross section. The slotted end of each flag is placed around the gage section of the specimen and secured using a room-temperature adhesive. The room-temperature adhesive holds the flags in position during subsequent handling and will burn off as the temperature is raised to the test temperature, but the room-temperature adhesive shall not react with the specimen during heating and subsequent burn-off. At this point the flags are held in place by friction. At elevated temperatures, oxidation secures the flags firmly to the gage section. Flag motion can then be monitored by using either a traveling microscope or a laser.

9.1.4.2 *Contacting Method*—Setting of the initial gage length for a contacting extensometer depends on the extension measurement method (capacitance based, strain gage based, or other), following the manufacturer's set-up procedures. The extensometer probes with knife-edge tips are positioned in contact with the specimen and typically held in place with a light (0.1 to 1.0 N) force. At elevated temperatures, oxidation at the probe-specimen interface minimizes slippage. Stability of the extensometer system shall be determined and reported for time durations comparable to actual tests.

9.2 *Temperature Control*—Form the thermocouple bead in accordance with the Preparation of Thermocouple Measuring Junctions.[5] Generally, noble-metal (for example, Pt or Rh) thermocouples shall not be attached directly to CFCC materials due to chemical incompatibility. The thermocouple junction may be brought close to the specimen (3 to 6 mm) and shielded. Shielding may be omitted if, for a particular furnace, the difference in indicated temperature from an unshielded bead and a bead inserted in a hole in the specimen has been shown to be less than one half the variation listed in 9.2.2. The bead shall be as small as possible and there shall be no shorting of the circuit (such as could occur from twisted wire behind the bead). Use ceramic insulators on the thermocouples in the hot zone. If some other electrical insulation material is used in the hot zone, it shall be carefully checked to determine whether the electrical insulating properties are maintained at higher temperatures.[6]

9.2.1 *Number of Required Thermocouples*— When the length of the specimen gage section is 25 to 50 mm, employ at least two thermocouples, one near each end of the gage section. For gage lengths of >50 mm, add a third thermocouple near the center of the gage section length.

9.2.2 *Temperature Limits*—For the duration of the test, the difference between the indicated temperature and the nominal test temperature shall not exceed the following limits:

Test Temperature	Variation
<1273 K	±3 K
≥1273 K	±6 K

In addition, temperature variation within the uniformly heated gage section shall not exceed the following, per 25 mm of gage section length:

Test Temperature	Variation
<773 K	±1 K
≥773 K	±1 % of the test temperature in degrees Kelvin

9.2.3 The term "indicated temperature" means the temperature that is indicated by the temperature measuring device using good quality pyrometric practice. It is recognized that true temperature may vary more than the indicated temperature. The permissible indicated temperature variations of 9.2.2 are not to be construed as minimizing the importance of good pyrometric practice and precise temperature control. All laboratories shall keep both indicated and true temperature variations as small as practicable. It is recognized that in view of the dependency of creep deformation of materials on temperature, close temperature measurement is necessary. The limits prescribed represent ranges that are common practice.

9.2.4 Temperature overshoots during heating shall not exceed the limits stated in 9.2.2. The heating characteristics of the furnace and the temperature control system shall be studied to determine the power input, temperature set point, proportioning control adjustment, and control-thermocouple placement to limit transient temperature overshoots. It may be desirable to stabilize the furnace at a temperature 10 to 25 K less than the nominal test temperature before making the final adjustments. Report any temperature overshoots with details of magnitude and duration.

9.2.5 *Temperature Rates and Hold Time*— The rate at which temperature can be increased from ambient to the final test temperature depends on many factors, such as heating system, temperature controller, test material, and test environment. Limiting time at the test temperature will minimize oxidation or time-dependent thermal degradation. In addition, some materials experience so-called oxidation due to low-temperature chemical instabilities which occur at intermediate temperatures. With these materials, the temperature ramp shall be as rapid as possible to minimize the exposure time to these intermediate temperatures. Generally, good results have been obtained for heating rates in which the specimen temperature is ramped from ambient to the test temperature in ≈30 min. The hold time at temperature prior to the start of the test shall be governed by the time necessary to ensure that the specimen and the output of the extensometer (if this is used to monitor strain during the test) have reached equilibrium and that the temperature can be maintained within the limits specified in 9.2.2. Report both the time to attain test temperature and the time at temperature before loading.

NOTE 3—Some CFCCs rely on the formation of oxide layers or on the flow of low viscosity phases for sealing and protecting the interior of the composite by preventing the ingression of the service environment at elevated temperatures. However, severe environmental degradation of some CFCCs has been documented (at temperatures as low as 573 K) when the service environment is allowed to ingress to the interior of the composite at temperatures where the formation of a protective oxide layer or the flow of glassy coatings is inhibited. This is particularly true for CFCCs that rely on the integrity of C and BN fiber coatings to promote fiber debonding and sliding, two of the micromechanical mechanisms responsible for their tough behavior.

[5] See *1982 Annual Book of ASTM Standards*, Part 44, Related Materials section.

[6] Steen, M., and Bressers, J., "A Facility for Uniaxial Testing of CMCs at High Temperatures," *High Temp. Chem. Processes* , 3, 1994, pp. 263–271.

9.3 *Conducting the Test*:

9.3.1 *Mounting the Specimen*—Each grip interface and specimen geometry described in Section 8 will require a unique procedure for mounting the specimen in the load train. If special components are required for each test, these shall be identified and noted in the test report. Mark the unheated part of the specimen with an indelible marker as to top, bottom, and front (side facing the operator) in relation to the test machine.

9.3.2 *Preparations for Testing*—Set the test mode and test rate on the test machine. Pre-load the specimen to remove the slack from the load train. The amount of pre-load which shall not exceed 10 % of the test load will depend on the material and tensile specimen geometry and shall be reported for each situation. Ready the autograph data acquisition systems for data logging. Begin recording furnace temperature when furnace heating is initiated and continue recording until the completion of the test. Maintain a constant minimal force in the load train to allow for the thermal expansion of the system during specimen heat up. It is recommended to use a test machine that allows for the control of the load during heating up and during mechanical loading up to the test stress. As indicated in 5.5, the loading rate will have a profound effect on the rate of load redistribution among the CFCC constituents depending on their relative creep resistance and on other effects such as matrix cracking for example. While different materials have different properties and therefore will respond differently to different loading rates, typical loading rates of 0.01 to 10 MPa/s may be used as starting points to investigate the effect of loading rate on the creep and creep rupture behavior of CFCCs. To investigate this effect, the use of dead weight loading configurations is discouraged since it is impractical to use such loading configurations to achieve a controlled loading rate.

9.3.2.1 Depending on the extensometer, it may be mounted to either (*1*) cold (ambient temperature) or (*2*) hot (elevated-temperature) specimens. (*1*) If the extensometer is mounted to a cold specimen, mount the extensometer on the specimen gage section at ambient temperature and zero the output. Enclose the specimen in the elevated-temperature furnace and lightly pack refractory insulation to seal the specimen and furnace. Do not pack the refractory overly tight so as to restrict the extensometer arms or pull rods or to introduce extraneous lateral or axial loads. Heat the specimen to the test temperature at the prescribed rate and hold constant at temperature until the specimen has reached thermal equilibrium. When the specimen has reached thermal equilibrium, re-zero the extensometer before conducting the test. (*2*) If the extensometer is to be mounted to a hot specimen, enclose the specimen in the elevated temperature furnace and lightly pack refractory insulation to seal the specimen and furnace. Be sure that the insulation is not packed overly tight so as to restrict the extensometer arms or pull rods or to introduce extraneous lateral or axial loads. Heat the specimen to the test temperature at the prescribed heating rate and hold constant at temperature until the specimen reaches thermal equilibrium. Mount the extensometer on the specimen gage section and zero the output. When the arms of the extensometer have reached thermal equilibrium, re-zero the extensometer before conducting the test.

9.3.3 *Conducting the Test*—Initiate data collection. Load the specimen at the prescribed loading rate to the test stress. As indicated in 5.5, loading rates can have a profound effect on the creep behavior and creep life of CFCCs. These effects are most evident when the rate of redistribution of internal stresses in the CFCC is comparable to the loading rate. At least obtain a record of strain versus time during the test. The test is completed when the specimen fails (that is, when it is unable to sustain the test load) or when the test is interrupted after a prescribed period of time. The rate of data collection shall reflect the logarithmic nature of most creep processes by taking more frequent readings at the beginning of the test when the rate of deformation is usually fastest. The interval for data collection shall not be more than 24 h or 0.1 % of the estimated duration of the test.

9.3.3.1 Creep tests are often interrupted after a prescribed period of time to determine the effect of creep on the tensile strength of CFCCs for example. The retained tensile strength can be reported either at the creep test temperature or at a different temperature. To determine the retained strength at the creep test temperature, unload the specimen at a prescribed unloading rate being aware, as indicated in 5.5 and 5.6, that the rate of mechanical unloading will have a profound effect on the redistribution of internal stresses and on the rate of creep recovery of the CFCC. As such, the unloading rate shall be chosen to either avoid or reveal rate effects on the retained strength by unloading at a sufficiently rapid or slow rate, respectively. After complete unloading, reload the specimen at a monotonic rate until failure. Follow Test Method C 1275 for details in the determination of the monotonic tensile strength of CFCCs. To determine the retained tensile strength at a temperature different from the creep test temperature, unload the specimen at a prescribed unloading rate to a minimal load level, being aware, as indicated in 5.5 and 5.6, that the rate of mechanical unloading will have a profound effect on the redistribution of internal stresses and on the rate of creep recovery. As such, the unloading rate shall be chosen to either avoid or reveal rate effects on the retained strength by unloading at a sufficiently rapid or slow rate respectively. Change the furnace temperature to the temperature at which the retained strength has to be determined. During specimen heating/cooling, maintain a constant minimal force in the load train to allow for the thermal expansion/contraction of the load train. Allow the specimen to reach thermal equilibrium at the new temperature and then load the specimen at a monotonic rate until failure. Follow Test Method C 1275 for details in the determination of the monotonic tensile strength of CFCCs. In all cases the complete thermal and mechanical schedule of the test shall be reported.

9.3.4 Determine the ambient temperature and relative humidity at the start and end of the test in accordance with Test Method E 337.

9.3.5 *Post-Test Dimensions*—Measure and report the gage section cross-sectional dimensions at the fracture location to 0.1 mm if the gage section has not been overly fragmented by

the fracture process. If an exact measure of the cross-sectional dimensions cannot be made due to fragmentation then the average dimensions measured in 9.1 shall be used.

9.3.5.1 Measure and report the fracture location relative to the midpoint of the gage section.

9.3.5.2 The time-to-failure of specimens fracturing outside the gage section (or outside the extensometer gage length of straight-sided specimens) are considered anomalous and can be used only as censored tests. However, the strain versus time recording can be considered representative of the creep behavior of the material under the test conditions.

9.3.5.3 Visual examination and light microscopy shall be conducted to determine the mode and type of fracture (that is, brittle or fibrous). In addition, although quantitative fractographic measurements are beyond the scope of this test method, observations can be made of the length of fiber pullout, orientation of fracture plane, degree of interlaminar fracture, and other pertinent details of the fracture surface.

9.4 *Fractography*—Fractographic examination of each failed specimen is recommended to characterize the fracture behavior of CFCCs. It should be clearly noted in the test report if a fractographic analysis was performed.

10. Calculation of Results

10.1 *General*—Various types of CFCC materials, due to the nature of their constituents, processing routes, and prior mechanical history, may exhibit vastly different stress-strain-time responses. Therefore, interpretation of the test results will depend on the type of response exhibited. The formula for determining the stress applied for a given load is as follows:

$$\sigma = P/A \quad (1)$$

where P is the load and A is the initial cross-sectional area of the specimen calculated as the product of the specimen width and thickness. The isothermal creep strain of the specimen at any time is determined as follows:

$$\epsilon = (l - l_o)/l_o \quad (2)$$

where l is the instantaneous gage length, as determined with an extensometer (see 9.3) and l_o is the gage length at the start of the creep test (determined also by the extensometer). The isothermal creep strain is comprised by time-independent elastic and inelastic components and a time-dependent component. The time-independent elastic and inelastic components may be determined from a tensile stress-strain curve obtained under monotonic mechanical loading at a very fast loading rate. The rate of creep deformation at any time is determined by the instantaneous derivative of the creep strain versus time curve.

11. Report

11.1 Report the following information:

11.1.1 Date and location of testing equipment.

11.1.2 Test specimen geometry (with engineering drawing), gripping procedure, mode of load train alignment, and results from load train alignment.

11.1.3 A drawing or sketch of the type and configuration of the test machine. If a commercial test machine is used, the manufacturer and model number will suffice.

11.1.4 All relevant data such as vintage and identification, with emphasis on the date of manufacture and a short description of reinforcement type, fiber architecture, fiber volume fraction, and bulk density.

11.1.5 Description of the method of specimen preparation including all stages of machining.

11.1.6 Test environment including test temperature and atmosphere, mode of temperature measurements, ambient relative humidity, and room temperature.

11.1.7 Complete thermomechanical schedule including loading/unloading rates, heating/cooling rates, and stress and temperature levels.

11.1.8 Plot of stress versus strain during the loading/unloading stages, plot of the creep strain versus time, and a plot of the creep strain rate versus time for each test.

11.1.9 In the event of specimen failure, report the test life of the specimen in minutes and hours and the creep strain at failure. If the test was interrupted and the specimen was fast fractured to determine the retained strength, report the duration of the creep test, the rate of unloading from the creep stress, and the monotonic loading rate and test mode for the determination of the retained strength (that is, load, strain or displacement control and loading rate). For report of retained strength results, follow Test Method C 1275.

11.1.10 Appearance of specimen after fracture (for example, formation of oxide layers, fracture surface, fiber pullout (or lack of), cracks in the matrix, etc.).

12. Precision and Bias

12.1 Because of the nature of the materials and lack of a wide database on a variety of applicable CFCCs, no definitive statement can be made at this time concerning precision and bias of the test methods herein.

13. Keywords

13.1 ceramic matrix composite; CFCC; continuous fiber ceramic composite; creep rupture; creep test; elevated temperature