# Standard Test Method for Monotonic Tensile Strength Testing of Continuous Fiber-Reinforced Advanced Ceramics With Solid Rectangular Cross-Section Specimens at Elevated Temperatures<sup>1</sup>

This standard is issued under the fixed designation C 1359; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon  $(\epsilon)$  indicates an editorial change since the last revision or reapproval.

### 1. Scope

- 1.1 This test method covers the determination of tensile strength including stress-strain behavior under monotonic uniaxial loading of continuous fiber-reinforced advanced ceramics at elevated temperatures. This test method addresses, but is not restricted to, various suggested test specimen geometries as listed in the appendix. In addition, specimen fabrication methods, testing modes (load, displacement, or strain control), testing rates (load rate, stress rate, displacement rate, or strain rate), allowable bending, temperature control, temperature gradients, and data collection and reporting procedures are addressed. Tensile strength as used in this test method refers to the tensile strength obtained under monotonic uniaxial loading where monotonic refers to a continuous nonstop test rate with no reversals from test initiation to final fracture.
- 1.2 This test method applies primarily to advanced ceramic matrix composites with continuous fiber reinforcement: unidirectional (1-D), bi-directional (2-D), and tri-directional (3-D) or other multi-directional reinforcements. In addition, this test method may also be used with glass (amorphous) matrix composites with 1-D, 2-D, 3-D and other multi-directional continuous fiber reinforcements. This test method does not directly address discontinuous fiber-reinforced, whisker-reinforced, or particulate-reinforced ceramics, although the test methods detailed here may be equally applicable to these composites.
- 1.3 The values stated in SI units are to be regarded as the standard and are in accordance with Practice E 380.
- 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. Refer to Section 7 for specific precautions.

### 2. Referenced Documents

2.1 ASTM Standards:

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- C 1145 Terminology of Advanced Ceramics<sup>2</sup>
- D 3379 Test Method for Tensile Strength and Young's Modulus for High-Modulus Single-Filament Materials<sup>3</sup>
- D 3878 Terminology of High Modulus Reinforcing Fibers and Their Composites<sup>3</sup>
- E 4 Practices for Force Verification of Testing Machines<sup>4</sup>
- E 6 Terminology Relating to Methods of Mechanical Testing<sup>4</sup>
- E 21 Practice for Elevated Temperature Tension Tests of Metallic Materials<sup>4</sup>
- E 83 Practice for Verification and Classification of Extensometers<sup>4</sup>
- E 220 Test Method for Calibration of Thermocouples by Comparison Techniques<sup>5</sup>
- E 337 Test Method for Measuring Humidity with Psychrometer (the Measurement of Wet-and Dry-Bulb Temperatures)<sup>6</sup>
- E 380 Practice for Use of International System of Units (SI) (the Modernized Metric System)<sup>7</sup>
- E 1012 Practice for Verification of Specimen Alignment Under Tensile Loading<sup>4</sup>

# 3. Terminology

- 3.1 Definitions:
- 3.1.1 Definitions of terms relating to tensile testing, advanced ceramics, fiber-reinforced composites as they appear in Terminology E 6, Terminology C 1145, and Terminology D 3878, respectively, apply to the terms used in this test method. Pertinent definitions are shown in the following with the appropriate source given in parentheses. Additional terms used in conjunction with this test method are defined in 3.2.
  - 3.2 Definitions of Terms Specific to This Standard:
- 3.2.1 advanced ceramic, n—a highly engineered, high-performance predominately nonmetallic, inorganic, ceramic material having specific functional attributes. (See Terminology C 1145.)
  - 3.2.2 axial strain  $[LL^{-1}]$ , n—the average longitudinal

 $<sup>^{\</sup>rm 1}$  This test method is under the jurisdiction of ASTM Committee C-28 on Advanced Ceramics and is the direct responsibility of Subcommittee C28.07 on Ceramic Matrix Composites.

<sup>&</sup>lt;sup>2</sup> Annual Book of ASTM Standards, Vol 15.01.

<sup>&</sup>lt;sup>3</sup> Annual Book of ASTM Standards, Vol 15.03.

<sup>&</sup>lt;sup>4</sup> Annual Book of ASTM Standards, Vol 03.01.

Annual Book of ASTM Standards, Vol 14.03.
 Annual Book of ASTM Standards, Vol 11.03.

<sup>&</sup>lt;sup>7</sup> Annual Book of ASTM Standards, Vol 14.02.

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strains measured at the surface on opposite sides of the longitudinal axis of symmetry of the specimen by two strainsensing devices located at the mid length of the reduced section. (See Practice E 1012.)

- 3.2.3 bending strain  $[LL^{-1}]$ , n—the difference between the strain at the surface and the axial strain. In general, the bending strain varies from point to point around and along the reduced section of the specimen. (See Practice E 1012.)
- 3.2.4 *breaking load [F]*, *n*—the load at which fracture occurs. (See Terminology E 6.)
- 3.2.5 ceramic matrix composite, n—a material consisting of two or more materials (insoluble in one another), in which the major, continuous component (matrix component) is a ceramic, while the secondary component/s (reinforcing component) may be ceramic, glass-ceramic, glass, metal, or organic in nature. These components are combined on a macroscale to form a useful engineering material possessing certain properties or behavior not possessed by the individual constituents.
- 3.2.6 continuous fiber-reinforced ceramic matrix composite (CFCC), n—a ceramic matrix composite in which the reinforcing phase consists of a continuous fiber, continuous yarn, or a woven fabric.
- 3.2.7 fracture strength  $[FL^{-2}]$ , n—the tensile stress that 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. (See Terminology E 6.)
- 3.2.7.1 *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.
- 3.2.8 gage length [L], n—the original length of that portion of the specimen over which strain or change of length is determined. (See Terminology E 6.)
- 3.2.9 matrix-cracking stress  $[FL^{-2}]$ , n—the applied tensile stress at which the matrix cracks into a series of roughly parallel blocks normal to the tensile stress.
- 3.2.9.1 *Discussion*—In some cases, the matrix cracking stress may be indicated on the stress-strain curve by deviation from linearity (proportional limit) or incremental drops in the stress with increasing strain. In other cases, especially with materials which do not possess a linear portion of the stress-strain curve, the matrix cracking stress may be indicated as the first stress at which a permanent offset strain is detected in the unloading stress-strain (elastic limit) curve.
- 3.2.10 modulus of elasticity  $[FL^{-2}]$ , n—the ratio of stress to corresponding strain below the proportional limit. (See Terminology E 6.)
- 3.2.11 *modulus of resilience* [FLL<sup>-3</sup>], *n*—strain energy per unit volume required to elastically stress the material from zero to the proportional limit indicating the ability of the material to absorb energy when deformed elastically and return it when unloaded.
- 3.2.12 modulus of toughness [FLL<sup>-3</sup>], n—strain energy per unit volume required to stress the material from zero to final fracture indicating the ability of the material to absorb energy beyond the elastic range (that is, damage tolerance of the material).
  - 3.2.12.1 *Discussion*—The modulus of toughness can also be

- referred to as the cumulative damage energy and as such is regarded as an indication of the ability of the material to sustain damage rather than as a material property. Fracture mechanics methods for the characterization of CFCCs have not been developed. The determination of the modulus of toughness as provided in this test method for the characterization of the cumulative damage process in CFCCs may become obsolete when fracture mechanics methods for CFCCs become available.
- 3.2.13 proportional limit stress  $[FL^{-2}]$ , n—the greatest stress which a material is capable of sustaining without any deviation from proportionality of stress to strain (Hooke's law). (See Terminology E 6.)
- 3.2.13.1 *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.
- 3.2.14 *percent bending*, *n*—the bending strain times 100 divided by the axial strain. (See Practice E 1012.)
- 3.2.15 *slow crack growth*, *n*—sub critical crack growth (extension) that may result from, but is not restricted to, such mechanisms as environmentally-assisted stress corrosion or diffusive crack growth.
- 3.2.16 tensile strength  $[FL^{-2}]$ , n—the maximum tensile stress which a material is capable of sustaining. Tensile strength is calculated from the maximum load during a tension test carried to rupture and the original cross-sectional area of the specimen. (See Terminology E 6.)

# 4. Significance and Use

- 4.1 This test method may be used for material development, material comparison, quality assurance, characterization, reliability assessment, and design data generation.
- 4.2 Continuous fiber-reinforced ceramic matrix composites generally characterized by crystalline matrices and ceramic fiber reinforcements are candidate materials for structural applications requiring high degrees of wear and corrosion resistance, and elevated-temperature inherent damage tolerance (that is, toughness). In addition, continuous fiberreinforced glass (amorphous) matrix composites are candidate materials for similar but possibly less-demanding applications. Although flexural test methods are commonly used to evaluate strengths of monolithic advanced ceramics, the non-uniform stress distribution of the flexure specimen in addition to dissimilar mechanical behavior in tension and compression for CFCCs leads to ambiguity of interpretation of strength results obtained from flexure tests for CFCCs. Uniaxially-loaded tensile-strength tests provide information on mechanical behavior and strength for a uniformly stressed material.
- 4.3 Unlike monolithic advanced ceramics that fracture catastrophically from a single dominant flaw, CFCCs generally experience 'graceful' (that is, non-catastrophic, ductile-like stress-strain behavior) fracture from a cumulative damage process. Therefore, the volume of material subjected to a uniform tensile stress for a single uniaxially-loaded tensile test may not be as significant a factor in determining the ultimate

strengths of CFCCs. However, the need to test a statistically significant number of tensile specimens is not obviated. Therefore, because of the probabilistic nature of the strengths of the brittle fibers and matrices of CFCCs, a sufficient number of specimens at each testing condition is required for statistical analysis and design. Studies to determine the influence of specimen volume or surface area on strength distributions for CFCCs have not been completed. It should be noted that tensile strengths obtained using different recommended tensile specimen geometries with different volumes of material in the gage sections may be different due to these volume differences.

- 4.4 Tensile tests provide information on the strength and deformation of materials under uniaxial tensile stresses. Uniform stress states are required to effectively evaluate any non-linear stress-strain behavior that may develop as the result of cumulative damage processes (for example, matrix cracking, matrix/fiber debonding, fiber fracture, delamination, etc.) that may be influenced by testing mode, testing rate, effects of processing or combinations of constituent materials, environmental influences, or elevated temperatures. Some of these effects may be consequences of stress corrosion or sub critical (slow) crack growth that can be minimized by testing at sufficiently rapid rates as outlined in this test method.
- 4.5 The results of tensile tests of specimens fabricated to standardized dimensions from a particular material or selected portions of a part, or both, may not totally represent the strength and deformation properties of the entire, full-size end product or its in-service behavior in different environments or 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 the particular primary processing conditions and post-processing heat treatments.
- 4.7 The tensile behavior and strength of a CFCC are dependent on its inherent resistance to fracture, the presence of flaws, or damage accumulation processes, or both. Analysis of fracture surfaces and fractography, though beyond the scope of this test method, is recommended.

### 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 measured tensile strength. In particular, the behavior of materials susceptible to slow crack growth fracture will be strongly influenced by test environment, testing rate, and elevated temperature of the test. Conduct tests to evaluate the maximum strength potential of a material in inert environments or at sufficiently rapid testing rates, or both, to minimize slow crack growth effects. Conversely, conduct tests in environments or at test modes, or both, and rates representative of service conditions to evaluate material performance under use conditions. Monitor and report relative humidity (RH) and temperature when testing is conducted in uncontrolled ambient air with the intent of evaluating maximum strength potential. Testing at humidity levels > 65 % RH is not recommended.
- 5.2 Surface preparation of test specimens, although normally not considered a major concern in CFCCs, can introduce

fabrication flaws which may have pronounced effects on tensile mechanical properties and behavior (for example, shape and level of the resulting stress-strain curve, tensile strength and strain, proportional limit stress and strain, etc.). Machining damage introduced during specimen preparation can be either a random interfering factor in the determination of ultimate strength of pristine material (that is, increase frequency of surface-initiated fractures compared to volume-initiated 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 methods for surface preparation do not exist. 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). Final machining steps may, or may not negate machining damage introduced during the initial machining. Therefore, report specimen fabrication history since it may play an important role in the measured strength distributions.

- 5.3 Bending in uniaxial tensile tests can cause or promote non-uniform stress distributions with maximum stresses occurring at the specimen surface leading to non-representative fractures originating at surfaces or near geometrical transitions. Bending may be introduced from several sources including misaligned load trains, eccentric or misshaped specimens, and non-uniformly 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 non-uniform 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 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 initiate fracture in the vicinity of the grips.

### 6. Apparatus

- 6.1 Testing Machines—Machines used for tensile testing shall conform to Practices E 4. As defined in Practices E 4, loads used in determining tensile strength shall be accurate within  $\pm$  1 % at any load within the selected load range of the testing machine. A schematic showing pertinent features of the tensile testing apparatus is shown in Fig. 1.
  - 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 specimen. The brittle nature of the matrices of CFCCs requires a uniform interface between the grip components and the gripped section of the specimen. Line or

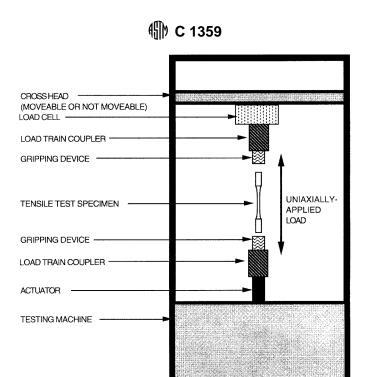
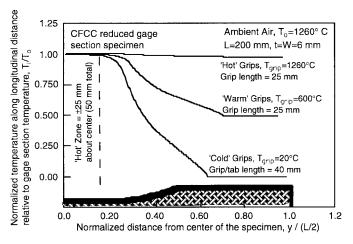


FIG. 1 Schematic Diagram of One Possible Apparatus for Conducting a Uniaxially-Loaded Tensile Test

point contacts and non-uniform pressure can produce Hertziantype 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 paragraphs. Uncooled grips located inside the heated zone are termed "hot grips" and generally produce almost no thermal gradient in the specimen but at the relative expense of grip materials of at least the same temperature capability as the test material and increased degradation of the grips due to exposure to the elevated-temperature oxidizing environment. 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 induce a mild thermal gradient in the specimen but at the relative expense of elevated-temperature alloys in the grips and increased degradation of the grips due to exposure to the elevated-temperature oxidizing environment. Cooled grips located outside the heated zone are termed "cold grips" and generally induce a steep thermal gradient in the specimen (as shown by example in Fig. 2) at a greater relative expense because of grip cooling equipment and allowances, although with the advantage of consistent alignment and little degradation from exposure to elevated temperatures.

Note 1—The expense of the cooling system for cold grips is balanced



Note 1—Shape is that of a quarter section of a face-loaded tensile specimen.

FIG. 2 Temperature Distributions in a Reduced Gage Section Specimen for Various Types of Gripping Arrangements

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 oxidizing environment. When grip cooling is employed, means should be provided to control the cooling medium to maximum fluctuations of 5 K (less than 1 K preferred) about a setpoint temperature (1)<sup>8</sup> 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 within  $\pm 5$  K (less than  $\pm 1$  K preferred) (1) so as to avoid introducing unequal thermal gradients and subsequent non uniaxial 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 required in 9.3.2.

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 applied by the test machine 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 uniform contact between the gripped section of the specimen and the grip faces and 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. Provide means to avoid such excessive stresses.

6.2.1.2 For flat specimens, face-loaded grips, either by direct lateral pressure grip faces (2) or by indirect wedge-type grip faces, act as the grip interface (3) as illustrated in Fig. 3 and Fig. 4, respectively. 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 configuration as shown in the appropriate specimen drawings.

6.2.1.3 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

<sup>8</sup> The boldface numbers given in parentheses refer to a list of references at the end of the text.

appears to be the most satisfactory. Keep the serrations 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 specimen.

6.2.1.4 Passive Grip Interfaces—Passive grip interfaces transmit the load applied by the test machine to the test specimen through a direct mechanical link. These mechanical links transmit the test loads to the specimen via 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.

6.2.1.5 For flat specimens, passive grips may act either through edge-loading via grip interfaces at the shoulders of the specimen shank (4) or by combinations of face-loading and pin loading via pins at holes in the gripped specimen head (5,6). 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 non-eccentric loading as shown in Fig. 5. In addition, moderately close tolerances are required for center-line coincidence and diameters of the pins and hole as indicated in Fig. 6.

6.2.1.6 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. In addition, wear of the grip cavity can be reduced by use of the thin brass sheets between the grip and specimen without adversely affecting specimen alignment.

6.2.1.7 The pins in the face/pin loaded grip are primarily for alignment purposes and load transmission. Secondary load transmission is through face-loading via 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.

6.2.1.8 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 fractions along interfaces particularly at geometric transitions or at discontinuities such as holes.

6.3 Load Train Couplers:

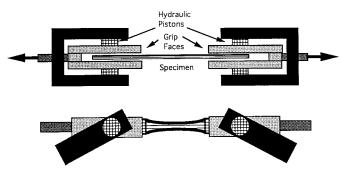


FIG. 3 Example of a Direct Lateral Pressure Grip Face for a Face-Loaded Grip Interface

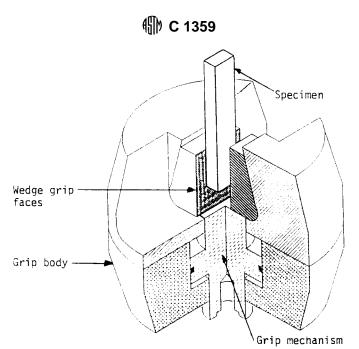


FIG. 4 Example of Indirect Wedge-Type Grip Faces for a Face-Loaded Grip Interface

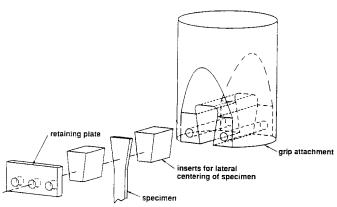


FIG. 5 Example of a Edge-Loaded, Passive Grip Interface (4)

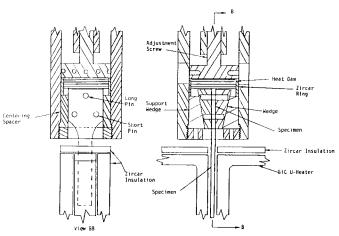


FIG. 6 Example of Pin/Face-Loaded Passive Grip Interface (5)

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 non-fixed as discussed in the following paragraphs. Use of well-aligned fixed or self-aligning non-fixed couplers does not automatically guarantee low bending in the gage section of the tensile specimen. Well-aligned fixed or

self-aligning non-fixed 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, verify alignment of the testing system at a minimum at the beginning and end of a test series unless the conditions for verifying alignment are otherwise met. An additional verification of alignment is recommended, although not required, at the middle of the test series. Use either a dummy or actual test specimen. Allowable bending requirements are discussed in 6.5. See Practice E 1012 for discussions of alignment and Appendix X1 for suggested procedures specific to this test method. 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 (for example, a test series composed of material A comprising ten specimens of geometry B tested at a fixed rate in strain control to final fracture in ambient air).

Note 2—Tensile specimens used for alignment verification should be equipped with a recommended eight separate longitudinal strain gages to determine bending contributions from both eccentric and angular misalignment of the grip heads. Ideally the verification specimen should 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, an alternate material (isotropic, homogeneous, continuous) with similar elastic modulus, elastic strain capability, and hardness to the test material may be used. In addition, dummy specimens used for alignment verification, should 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, pre-test alignment adjustment of the load train that remains constant for all subsequent tests or an in-situ, pre-test alignment of the load train which is conducted separately for each specimen and each test. Such devices (7,8) usually employ angularity and concentricity adjusters to accommodate inherent load-train misalignments. Regardless of which method is used, verify alignment verification as discussed in 6.3.1.1.
- 6.3.2.1 Fixed load-train couplers are preferred in the monotonic testing of CFCCs because of the fracture behavior in these materials. During the fracture process of CFCCs, the fixed coupler tends to hold the specimen in an aligned position and thus provides a continuous uniform stress across the remaining ligament of the gage section.
- 6.3.3 Non Fixed Load-Train Couplers—Non fixed couplers may incorporate devices which promote self-alignment of the load train during the movement of the crosshead or actuator. 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 (5,9,10) of such devices. Examples of two such devices are shown in Fig. 7. Although non-fixed load-train couplers are designed to be self-aligning and thus eliminate the

need to evaluate the bending in the specimen for each test, this alignment must be confirmed. Verify the operation of the couplers as discussed in 6.3.1.1.

- 6.3.3.1 Non-fixed load-train couplers are useful in testing of CFCCs at rapid test rates or in load control where the cumulative-damage fracture process may not be as macroscopically apparent. If the material exhibits such fracture behavior the self-aligning feature of the non-fixed coupler allows rotation of the gripped section of the specimen thus promoting a non-uniform stress in the remaining ligament of the gage section.
- 6.4 Strain Measurement—Determine strain at elevated temperatures by means of a suitable extensometer.
- 6.4.1 Extensometers used for tensile testing of CFCC specimens shall satisfy Practice E 83, Class B-1 requirements. Calibrate extensometers periodically in accordance with Practice E 83. For extensometers which mechanically contact the specimen, the contact shall not cause damage to the specimen surface. However, shallow grooves (0.025 to 0.051 mm deep) machined into the surfaces of CFCCs to prevent extensometer slippage have been shown to not have a detrimental effect on failure strengths at elevated temperatures (5). Choose extensometer contact probes which are chemically compatible with the test material (for example, alumina extensometer extensions and SiC CFCC are incompatible). In addition, support the weight of the extensometer so as not to introduce bending greater than that allowed in 6.5. Finally, configure the tips of the probes of contacting extensometers (for example, sharp, knife edges, or chisel tips) so as to minimize slippage.
- 6.5 Allowable Bending—Analytical and empirical studies (11) have concluded that for negligible effects on the estimates of the strength distribution parameters (for example, Weibull modulus,  $\hat{\mathbf{m}}$ , and characteristic strength,  $\hat{\sigma}_{\theta}$ ) of monolithic advanced ceramics, allowable percent bending as defined in Practice E 1012 should not exceed five. These conclusions (11) assume that tensile strength fractures are due to single fracture origins in the volume of the material, all tensile specimens experienced the same level of bending, and that Weibull modulus,  $\hat{\mathbf{m}}$ , was constant.
- 6.5.1 Similar studies of the effect of bending on the tensile strength distributions 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 bending at the onset of the cumulative fracture process (for example, matrix cracking stress) for specimens tested under this test method is five. Verify the testing system such that percent bending does not exceed five at a mean strain equal to either one half the anticipated strain at the onset of the cumulative fracture process (for example, matrix cracking stress) or a strain of 0.0005 (that is, 500 micro strain) whichever is greater. Unless all specimens are properly strain gaged and percent bending monitored until the onset of the cumulative fracture process, there will be no record of percent bending at the onset of fracture for each specimen. Therefore, verify the alignment of the testing system. See Practice E 1012 for discussions of alignment and Appendix X1 for suggested procedures specific to this test method.

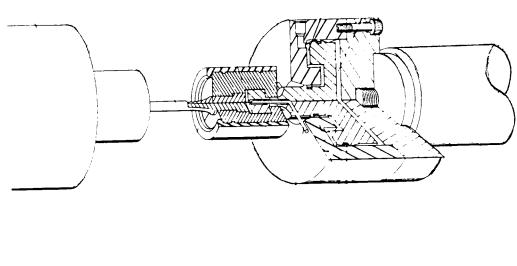


FIG. 7 Examples of Hydraulic, Self-Aligning, Non Fixed Load Train Couplers (9,10)

- 6.6 *Heating Apparatus*—The apparatus for, and method of, heating the specimens shall provide the temperature control necessary to satisfy the requirement of 9.3.2.
- 6.6.1 Heating can be by indirect electrical resistance (heating elements), direct induction, 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 3—Direct resistance heating is not recommended for heating CFCCs due to possible differences of the electrical resistances of the constituent materials that 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 9.3.2.
- 6.7.1 For test temperatures less than 2000 K, make primary temperature measurements with noble-metal thermocouples in conjunction with potentiometers, millivoltmeters, or electronic temperature controllers or readout units, or all of these. Such measurements are subject to two types of error as discussed in MNL 12 (12). Firstly, 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.
- 6.7.1.1 Measure temperature with thermocouples of known calibration (calibrated according to Test Method E 220). Calibrate representative thermocouples from each lot of wires used for making noble (for example, Pt or Rh/Pt) metal thermocouples. Except for relatively low temperatures of exposure, noble-metal thermocouples are eventually subject to error upon reuse, unless the depth of immersion and temperature gradients of the initial exposure are reproduced. Consequently, calibrate noble-metal thermocouples using representative thermocouples. Do not reuse degraded noble-metal thermocouples without proper treatment. This treatment includes clipping back the wire exposed to the hot zone, rewelding a thermocouple bead, and properly annealing the rewelded thermocouple bead and wire. Any reuse of noble-metal thermocouples (except after relatively low-temperature use) without this precautionary treatment shall be accompanied by recalibration data demonstrating that calibration of the temperature reading system was not unduly affected by the conditions of exposure.
- 6.7.1.2 Measurement of the drift in calibration of thermocouples during use is difficult. When drift is a problem during tests, devise a method to check the readings of the thermocouples on the specimen during the test. For reliable calibration of thermocouples after use, reproduce the temperature gradient of the test furnace during the recalibration.
- 6.7.1.3 Thermocouples containing Pt are also subject to degradation in the presence of silicon and silicon-containing compounds. Platinum silicides may form leading to several

possible outcomes. One outcome is the embrittlement of the noble-metal thermocouple tips and their eventual degradation and breakage. Another outcome is the degradation of the silicon-containing material (for example, test specimen, furnace heating elements, or refractory furnace materials). In all cases, do not allow platinum containing materials to contact silicon containing materials. In particular, do not allow noblemetal thermocouples to contact silicon-based test materials (for example, SiC or  $\mathrm{Si}_3\mathrm{N}_4$ ). In some cases (for example, when using SiC heating elements), it is advisable to use ceramic-shielded noble-metal thermocouples to avoid the reaction of the Pt-alloy thermocouples with the SiO gas generated by the volatilization of the  $\mathrm{SiO}_2$  protective layers of SiC heating elements.

- 6.7.1.4 Calibrate temperature-measuring, controlling, and recording instruments versus a secondary standard, such as precision potentiometer, optical pyrometer, or black-body thyristor. Check lead-wire error with the lead wires in place as they normally are used.
- 6.7.2 For test temperatures greater than 2000 K, less-common temperature measurement devices such as thermocouples of elevated-temperature, non noble-metal alloys (for example, W-Re) or optical pyrometry may be used. Since widely-recognized standards do not exist for these less-common devices, report the type of measurement device, its method of calibration, and its accuracy and precision.
- 6.8 Data Acquisition—At a minimum, obtain an autographic record of applied load and gage section elongation or strain versus time. 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, use an analog chart recorder or plotter 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 to within  $\pm 1.0$  % of the selected range for the testing system including readout unit, as specified in Practices E 4, and should have a minimum data acquisition rate of 10 Hz with a response of 50 Hz deemed more than sufficient.
- 6.8.1 Record strain or elongation, or both, of the gage section either similarly to the load or as independent variables of load. Cross-head displacement of the test machine may also be recorded but should 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 At a minimum, record temperature as single points at the initiation and completion of the actual test. However, temperature can also be recorded similarly to load and strain except the record can begin 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 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 using dimension measuring devices with accuracies of 0.01 mm.

### 7. Precautionary Statement

7.1 During the conduct of this test method, the possibility of flying fragments of broken test material may be great. 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 safety as well as later fractographic reconstruction and analysis is recommended.

7.2 Exposed fibers at the edges of CFCC specimens present a hazard due to the sharpness and brittleness of the ceramic fiber. Inform all persons required to handle these materials of such conditions and the proper handling techniques.

### 8. Test Specimen

8.1 Test Specimen Geometry:

8.1.1 General—The geometry of tensile test specimens is dependent on the ultimate use of the tensile strength data. For example, if the tensile strength 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 via a particular processing route, then the size of the specimen and resulting gage section will reflect the desired volume or surface area 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 paragraphs discuss the more common, and thus proven, of these specimen geometries although any geometry is acceptable if it meets the gripping, fracture location, bending, and temperature profile requirements of this test method. Deviations from the recommended geometries may be necessary depending upon the particular CFCC being evaluated. Conduct stress analyses of untried specimens to ensure that stress concentrations which can lead to undesired fractures outside the gage sections do not exist. 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 tensile 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, analyze untried test configurations simultaneously for both loading-induced stress gradients and thermally-induced temperature gradients to ascertain any adverse interactions.

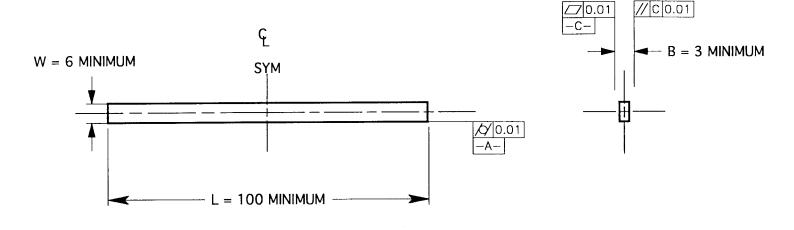
8.1.1.2 Specimens with contoured gage sections (transition radii of > 50 mm) are preferred to promote the tensile stresses with the greatest values in the uniformly-stressed gage section (13) while minimizing the stress concentration due to the geometrical transition of the radius. However, in certain 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 (that is, non-contoured) specimens as shown in Fig. 8, may be required for determining the tensile strength behavior of the CFCC. 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 in any of the methods discussed here although active gripping systems are recommended for minimizing non-gage section fractures.

8.1.2 Edge-Loaded Flat Tensile Specimens—Fig. 9 and Fig. 10 show examples of edge-loaded specimens which utilize the lateral compressive stresses developed at the specimen/grip interface at the gripped section as the specimen is pulled into the wedge of the grip (4). This type of geometry has been successfully employed for the evaluation of 1-D, 2-D, and 3-D CFCCs. Of particular concern with this geometry is the proper and consistent angle of the edge-loaded shank as shown in Fig. 9 and Fig. 10. Thus, the edge-loaded geometry may require somewhat intensive fabrication and inspection procedures.

8.1.3 Face-Loaded Flat Tensile Specimens—Fig. 11, Fig. 12 and Fig. 13 show examples of face-loaded specimens which exploit 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 straightsided (that is, non-contoured) specimens, end tabs may be required to provide a compliant layer for gripping. Balanced 0/90° cross-ply tabs made from unidirectional non-woven E-glass have proven to be satisfactory for certain fiberreinforced polymers. For CFCCs, tab materials comprised of fiberglass reinforced epoxy, polymethylene resins (PMR), or carbon fiber-reinforced resins have been used successfully (13). 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 within  $\pm 10$  % of bulk elastic modulus of the CFCC) with the CFCC material being tested. Each beveled tab (bevel angle < 15°) should 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 ( $\geq 10$  to 20 %) of fractures within one specimen width of the tab shall be cause to re-examine the tab materials and configuration, gripping method and adhesive, and to make necessary adjustments to promote fracture within the gage section. Fig. 14 shows an example of tab design which has been used successfully with CFCCs (13). Take care to ensure that both the adhesive and tab material are capable of use at the temperature that might occur in the grip region.



NOTE: 1) MINIMUM L = 100 mm WITH 25 mm GAGE SECTION. MINIMUM W = 6 mm.

2) SURFACE FINISH 0.5- 1.0 µm ALL OVER EXCEPT END FACES WHICH MAY BE 1.0-2.0 µm.

3) FINAL GRIND OF GAGE SECTION TO BE LONGITUDINAL

Tensile Specimen for CFCCs

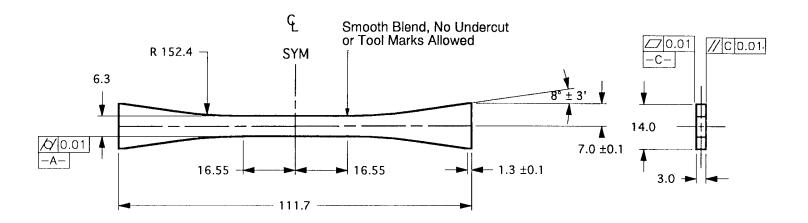
mm X.X = 0.1, X.XX = 0.01, X.XXX = 0.001 SCALE: NTS

Note 1—Illustration not intended to be an engineering or production drawing, or both.

FIG. 8 Example of Straight-Sided Specimen Geometry







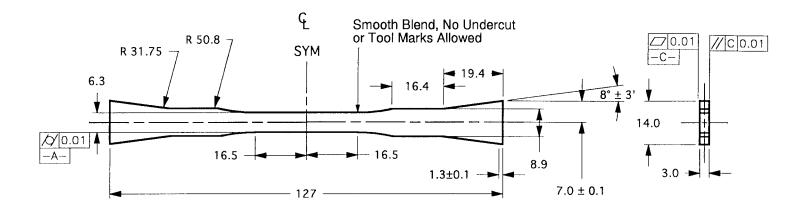
NOTE: 1) FINAL GRIND OF GAGE SECTION TO BE LONGITUDINAL

Tensile Specimen for CFCCs	
mm X.X = 0.1, X.XX = 0.01, X.XXX = 0.001 SCALE: NTS	

Note 1—Illustration not intended to be an engineering or production drawing, or both.

FIG. 9 Example of a Contoured, Edge-Loaded Specimen Geometry (4)





NOTE: 1) FINAL GRIND OF GAGE SECTION TO BE LONGITUDINAL

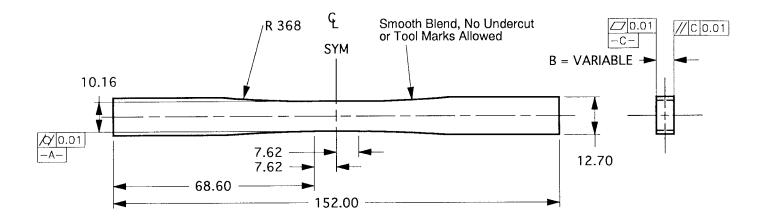
Tensile Specimen for CFCCs

mm X.X = 0.1, X.XX = 0.01, X.XXX = 0.001

SCALE: NTS

Note 1—Illustration not intended to be an engineering or production drawing, or both.

FIG. 10 Example of a Contoured, Edge-Loaded Specimen Geometry (4)



NOTE: 1) SURFACE FINISH 0.5- 1.0 µm ALL OVER EXCEPT END FACES WHICH MAY BE 1.0-2.0 µm.
2) FINAL GRIND OF GAGE SECTION TO BE

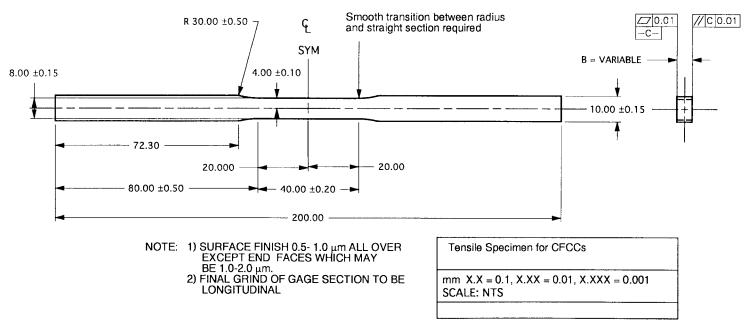
LONGITUDINAL

Tensile Specimen for CFCCs

mm X.X = 0.1, X.XX = 0.01, X.XXX = 0.001SCALE: NTS

Note 1—Illustration not intended to be an engineering or production drawing, or both.

FIG. 11 Example of a Contoured, Face-Loaded Specimen Geometry (13)



Note 1—Illustration not intended to be an engineering or production drawing, or both. FIG. 12 Example of a Contoured, Face-Loaded Specimen Geometry

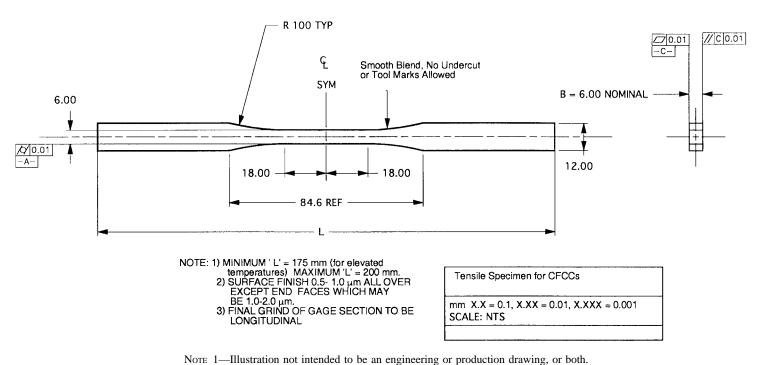
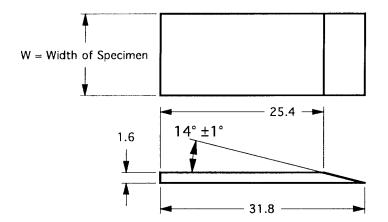


FIG. 13 Example of a Contoured, Face-Loaded Specimen Geometry





### TOWARD LONGITUDINAL MIDPOINT OF SPECIMEN



NOTE: 1) SURFACE FINISH 0.5- 1.0 µm ALL OVER EXCEPT END FACES WHICH MAY BE 1.0-2.0 µm.
2) FINAL GRIND OF GAGE SECTION TO BE

LONGITUDINAL

3) ANGLE OF BEVEL SHOULD BE ≤15°

Tabs for Tensile Specimen for CFCCs

mm X.X = 0.1, X.XX = 0.01, X.XXX = 0.001

**SCALE: NTS** 



Note 1—Illustration not intended to be an engineering or production drawing, or both.

FIG. 14 Example of a Bevelled Tab Successfully Used With Face-Loaded CFCC Tensile Specimens (13)

8.1.4 Pin/Face-Loaded Flat Tensile Specimens—The specimens shown in Fig. 15 and Fig. 16 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.

Note 4—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 strength data, use one of the following specimen preparation procedures. Regardless of the preparation procedure used, report sufficient details regarding the procedure to allow replication.
- 8.2.2 As-Fabricated—The tensile specimen shall 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 may possess rough surface textures and non-parallel edges and as such may cause excessive misalignment or be prone to non-gage section fractures, or both.
- 8.2.3 Application-Matched Machining—Finish the tensile specimen as close to the same surface/edge preparation as that applied to the component. Unless the process is proprietary, report specifics 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 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), use this procedure.
- 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 may be viewed as starting-point guidelines and a more stringent procedure may be necessary.
- 8.2.5.1 Perform all grinding or cutting with an ample supply of appropriate filtered coolant to keep the workpiece and grinding wheel constantly flooded and particles flushed. Grind in at least two stages, ranging from coarse to fine rate of material removal. Cut in one stage appropriate for the depth of cut.
- 8.2.5.2 Remove stock on the order of 0.03 mm per pass using diamond tools that have between 320 and 600 grit.

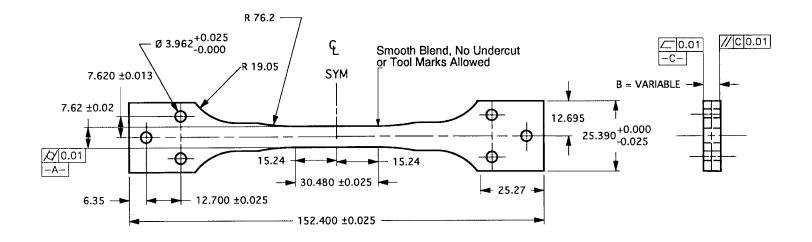
Remove equal stock from each face where applicable.

- 8.3 Handling Precaution—Exercise care in storing and handling finished specimens to avoid the introduction of random and severe flaws. In addition, pay attention to pre-test storage of specimens in controlled environments or desiccators to avoid unquantifiable environmental degradation of specimens prior to testing.
- 8.4 Number of Specimens—A minimum of five specimens is required for the purpose of estimating a mean. A greater number of specimens may be necessary if estimates regarding the form of the strength distribution are required. If material cost or specimen availability limit the number of tests to be conducted, fewer tests can be conducted to determine an indication of material properties.
- 8.5 *Valid Tests*—A valid individual test is one which meets all the following requirements: all the testing requirements of this test method, and failure occurs in the uniformly-stressed gage section unless those tests failing outside the gage section are interpreted as interrupted tests for the purpose of censored test analyses.

### 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 critical gage section area make these measurements 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 may be preferred when measuring specimens with rough or uneven surfaces. Report the measured dimensions and locations of the measurements for use in the calculation of the tensile stress. Use the average of the multiple measurements 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 procedures described in 9.1. 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, it is advisable to follow the procedures outlined in 9.1 for pretest measurements to ensure reliable measurements.
- 9.1.2 Conduct periodic, if not 100 %, inspection/ measurements of all specimens and specimen dimensions to ensure 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 at the equipment meets the specifications in 6.9. 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 the surface condition. Such methods as contacting profilometry can be used to determine surface roughness perpendicular to the tensile axis. When quantified, report surface roughness.





NOTE: 1) SURFACE FINISH 0.5- 1.0 µm ALL OVER EXCEPT END FACES WHICH MAY BE 1.0-2.0 µm.

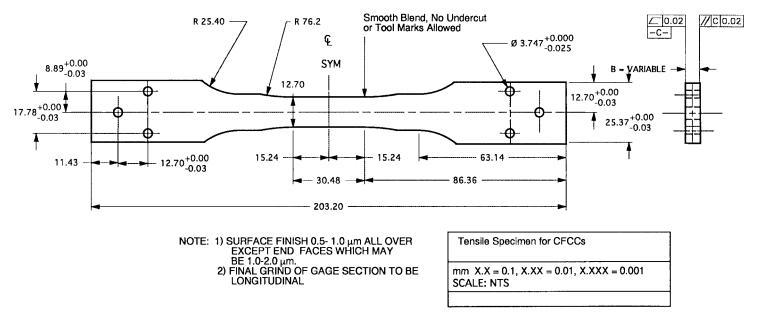
2) FINAL GRIND OF GAGE SECTION TO BE LONGITUDINAL

Tensile Specimen for CFCCs

mm X.X = 0.1, X.XX = 0.01, X.XXX = 0.001

STUDINAL SCALE: NTS

Note 1—Illustration not intended to be an engineering or production drawing, or both. FIG. 15 Example of a Contoured, Pin/Face-Loaded Specimen Geometry (5)



Note 1—Illustration not intended to be an engineering or production drawing, or both.

FIG. 16 Example of a Contoured, Pin/Face-Loaded Specimen Geometry (6)

9.2 Test Modes and Rates:

9.2.1 General—Test modes and rates can have distinct and strong influences on fracture behavior of advanced ceramics especially at elevated temperatures depending on test environment or condition of the specimen. Test modes may involve load, displacement, or strain control. Recommended rates of testing are intended to be sufficiently rapid to obtain the maximum possible tensile strength at fracture of the material. However, rates other than those recommended here may be used to evaluate rate effects. In all cases, report the test mode and rate.

Note 5—For monolithic advanced ceramics exhibiting linear elastic behavior, fracture is attributed to a weakest-link fracture mechanism generally attributed to stress-controlled fracture from Griffith-like flaws. Therefore, a load-controlled test, with load generally related directly to tensile stress, is the preferred test mode. However, in CFCCs the non-linear stress-strain behavior characteristic of the cumulative-damage fracture process of these materials indicates strain dependent behavior. Generally, displacement- or strain-controlled tests are employed in such cumulative damage or yielding deformation processes to prevent a 'run away' condition (that is, rapid uncontrolled deformation and fracture) characteristic of load or stress controlled tests. Thus, to elucidate the potential 'toughening' mechanisms under controlled fracture of the CFCC, displacement or strain control is preferred. However, for sufficiently rapid test rates, differences in the fracture process may not be noticeable and any of these test modes may be appropriate.

9.2.2 Strain Rate—Strain is the independent variable in non-linear analyses such as yielding. As such, strain rate is a method of controlling tests of deformation processes to avoid' runaway' conditions. For the linear elastic region of CFCCs, strain rate can be related to stress rate such that:

$$\dot{\epsilon} = \frac{d\epsilon}{dt} = \frac{\dot{\sigma}}{E} \tag{1}$$

where:

 $\dot{\epsilon}$  = strain rate in the specimen gage section, s<sup>-1</sup>,

 $\epsilon$  = strain in the specimen gage section,

t = time. s

σ = nominal stress rate in the specimen gage section, MPa/s, and.

E = elastic modulus of the CFCC, MPa.

Strain-controlled tests can be accomplished using an extensometer contacting the gage section of the specimen as the primary control transducer.

Note 6—Strain rates on the order of  $50 \times 10^{-6}$  to  $500 \times 10^{-6}$  s<sup>-1</sup> are recommended to minimize environmental effects when testing in ambient air. Alternatively, select strain rates to produce final fracture in 5 to 10 s so as to minimize environmental effects when testing in ambient air.

9.2.3 Displacement Rate—The size differences of each specimen geometry require a different loading rate for any given stress rate. As the specimen begins to fracture, the strain rate in the gage section of the specimen will change even though the rate of motion of the cross head remains constant. For this reason, displacement rate controlled tests can give only an approximate value of the imposed strain rate. Displacement mode is defined as the control of, or free-running displacement of, the test machine cross head. Thus, the displacement rate can be calculated as follows. Using the recommended (or desired) strain rate as detailed in 9.2.2, calculate the displacement rate for the linear elastic region of CFCCs only as:

$$\tilde{\delta} = \frac{d\delta}{dt} \approx \left(\frac{1}{k_m} + \frac{1}{k_s}\right) \dot{\epsilon} EA = \left(\frac{1}{k_m} + \frac{1}{k_s}\right) \dot{\sigma} A$$
 (2)

where:

= displacement rate of the cross head, mm/s,

 $\delta$  = cross-head displacement in units of mm,

 $k_m$  = stiffness of the test machine and load train (including the specimen ends and the grip interfaces), N/mm,

 $k_s$  = stiffness of the uniform gage section of the specimen,

E = elastic modulus of the material in units of MPa, and

A =cross-sectional area of the gage section.

Calculate the cross-sectional area, A, as A = w b for rectangular cross sections where w is the width of the gage section in units of mm, and b is the thickness of the gage section in units of mm.

Note 7—For L as the ungripped length of the specimen,  $k_s$  can be calculated as  $k_s$ = AE/L. The stiffness  $k_m$  can be determined as described in Test Method D 3379 by measuring the load-displacement curves for various specimen lengths. The plot of  $k_m$  (slope of load-displacement curve) versus specimen length is then extrapolated to zero to find the actual machine stiffness. Alternatively,  $k_m$  can be estimated using the manufacturer's value for frame stiffness as a starting point and decreasing this value as necessary to account for various links in the load train. If such a method is used, report the assumptions and methods for approximating  $k_m$ .

9.2.4 Load Rate—For materials which do not experience gross changes in cross-sectional area of the gage section, load rate can be directly related to stress rate and hence to the recommended (or desired) strain rate. For the linear elastic region of CFCCs, calculate load rate as:

$$\mathbf{\mathcal{C}} = \frac{dP}{dt} = \dot{\sigma}A \approx \dot{\epsilon}EA \tag{3}$$

where:

 $\dot{P}$  = required load rate, N/s, and

P = applied force, N.

Note 8—As the specimen begins to fracture, the strain rate in the gage section of the specimen will change even though the rate of load application remains constant. Stress rates > 35 to 50 MPa/s have been used with success (14) in tensile testing CFCCs to minimize the influence of environmental effects. If environmental effects apply for tensile strengths, then similar test rates should be chosen to obtain the greatest value of ultimate tensile strength. Alternately, select stress or load rates to produce final fracture in 5 to 10 s to minimize environmental effects when testing in ambient air.

9.2.5 Ramp Segments—Normally, tests are conducted in a single ramp function at a single test rate from zero load to the maximum load at fracture. However, in some instances multiple ramp segments might be employed. In these cases, a slow test rate is used to ramp from zero load to an intermediate load to allow time for removing "slack" (that is, loose and nontensioned) from the test system. The final ramp segment of the test is conducted from the intermediate load to the maximum load at fracture at the required (desired) test rate, although hold times are not allowed to avoid environmental effects. Report the type and time duration of the ramp.

9.3 *Temperature Control*—If thermocouples are used, form the thermocouple bead in accordance with Practice E 21. Do not attach noble-metal (for example, Pt or Rh) thermocouples

directly to CFCC materials due to possible chemical incompatibility. The thermocouple junction may be brought close to the specimen (3 to 6 mm) and shielded from thermal radiation in the furnace. 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 9.3.2. Make the bead as small as possible although there should 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, carefully check it to determine whether the electrical insulating properties are maintained at greater temperatures.

- 9.3.1 *Number of Required Thermocouples*—When the length of the specimen gage section is 25 to 50 mm and thermocouples are used, employ at least two thermocouples, one near each end of the gage section. For lengths of > 50 mm, add a third thermocouple near the center of the gage section length.
- 9.3.2 *Temperature Limits*—For the duration of the test do not permit the difference between the indicated temperature and the nominal test temperature to exceed the following limits:

$$\leq$$
 1273 K  $\pm$ 3 K  $\rightarrow$  1273 K  $+$ 6 K

9.3.2.1 In addition, temperature gradient within the uniformly-heated gage section shall not exceed the following:

$$\leq$$
 773 K  $\pm$ 5 K  $\Rightarrow$  773 K  $\pm$ 1 % of the test temperature (K)

- 9.3.3 The term "indicated temperature" means temperature that is indicated by the temperature measuring device using good quality pyrometric practice. True temperature may vary more than the indicated temperature. The permissible indicated temperature variations of 9.3.2 are not to be construed as minimizing the importance of good pyrometric practice and precise temperature control. All laboratories should keep both indicated and true temperature variations as small as practicable. In view of the extreme dependency of strength of materials on temperature, close temperature measurement is necessary. The limits prescribed represent ranges that are common practice.
- 9.3.4 Temperature overshoots during heating shall not exceed the following limits:

- 9.3.4.1 Study the heating characteristics of the furnace and the temperature control system 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.
- 9.3.5 *Temperature Rates and Hold Time*—The rate at which temperature can be increased from ambient to the test temperature depends on many factors, such as: heating system, temperature controller, test material, and test environment. The

hold time at temperature prior to the start of the test should be governed by the time necessary to ensure that the specimen has reached equilibrium, the time necessary to stabilize the strain-measurement device, and time necessary to ensure that temperature can be maintained within the limits specified in 9.3.2. This hold time should generally not exceed 30 min. Report both the time to attain test temperature and the time at temperature before loading.

Note 9—When tensile testing for intrinsic strength (that is, strength at temperature only and not degraded by effects of time at temperature), limit time at temperature to that necessary to equilibrate the specimen at the test temperature. 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 much less than upper limit elevated temperatures. In these materials, ramp the temperature as rapidly as possible to minimize the exposure time to these intermediate temperatures. Good results have been obtained for heating rates in which the specimen temperature is ramped from ambient to the test temperature in approximately 30 min.

- 9.4 Conducting the Tensile Test:
- 9.4.1 *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. Report any special components required for each test. Mark the unheated part of the specimen with an indelible marker as to top and bottom and front (side facing the operator) in relation to the test machine.
- 9.4.2 Preparations for Testing—Set the test mode and test rate on the test machine. Preload the specimen to remove the "slack" from the load train. For each situation, determine and report the amount of preload which will depend on the material and tensile specimen geometry. Heating of the specimen should be done at or near zero load in load control test mode if possible. Ready the autograph data acquisition systems for data logging. If desired, begin recording furnace temperature when furnace heating is initiated and continue recording until the completion of the test.

Note 10—Thermal expansion of the specimen during heating may lead to changes in alignment if the tensile preload is reduced or build up of axial compressive forces in a fixed actuator system that may damage the specimen if load control test mode is not employed. The preload should be sufficient to maintain load train alignment while in load control. If load control is not available, the actuator position can be adjusted as necessary during heat up to maintain a preload sufficient to hold the load train alignment.

- 9.4.2.1 Depending on the extensometer, mount it on the specimen either while the system is cold (ambient-temperature) or after the specimen has been heated to the test temperature (elevated-temperature) as detailed in the following paragraphs.
- 9.4.2.2 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. Be sure that the insulation is not packed overly tight so as to restrict the extensometer arms or pullrods 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.

9.4.2.3 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 pullrods 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 a desired temperature (usually near or at the test temperature). Mount the extensometer on the specimen gage section and zero the output. When the specimen has reached thermal equilibrium, re-zero the extensometer before conducting the test.

9.4.3 Conducting the Test—If test temperature is not being recorded continuously, record the test temperature at test initiation. Initiate the data acquisition. Initiate the test mode. After specimen fracture, disable the action of the test machine and the data collection of the data acquisition system. Record the breaking load with an accuracy of 1.0 % of the load range. Record test temperature at test completion. Cool the specimen and test apparatus to ambient temperature. Carefully remove the specimen from the grip interfaces. Take care not to damage the fracture surfaces by preventing them from contacting each other or other objects. Place the specimen along with any fragments from the gage section into a suitable, non-metallic container for later analysis.

9.4.4 Determine the ambient temperature and relative humidity in accordance with Test Method E 337.

9.4.5 Post-Test Dimensions—If necessary, measure and report gage section cross-sectional dimensions at the fracture location 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 use the average dimensions measured in 9.1.

9.4.5.1 Measure and report the fracture location relative to the midpoint of the gage section. Use the convention that the midpoint of the gage section is 0 mm with positive (+) measurements toward the top of the specimen as tested (and marked) and negative (-) measurements toward the bottom of the specimen as tested (and marked). For fracture surfaces which are not normal to the longitudinal axis the average fracture location may be reported. Record and report the orientation of the fracture and fracture locations.

Note 11—Results from specimens fracturing outside the uniformly stressed gage section are not recommended for use in the direct calculation of a mean tensile strength at fracture for the entire test set. Results from specimens fracturing outside the uniformly stressed gage section are considered anomalous and can be used only as censored tests (that is, specimens in which a tensile stress at least equal to that calculated by Eq 7 was sustained in the uniform gage section before the test was prematurely terminated by a non-gage section fracture). From a conservative standpoint in completing a required statistical sample (for example, n=5) for purposes of average strength, test one replacement specimen for each specimen which fractures outside the gage section.

9.5 Fractography—Conduct visual examination and light microscopy, if necessary, to determine the mode and type of fracture (that is, brittle or fibrous). In addition, although quantitatively beyond the scope of this test method, subjective

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. Fractographic examination of each failed specimen is recommended to characterize the fracture behavior of CFCCs.

#### 10. Calculation

10.1 General—Due to the nature of their constituents, processing routes, and prior mechanical history, various types of CFCC materials may exhibit different stress-strain responses as illustrated schematically in Fig. 17 (a), (b), (c). Therefore, interpretation of the test results will depend on the type of response exhibited. Points corresponding to the following calculated values are shown on the appropriate diagrams.

10.2 Engineering Stress—Calculate the engineering stress as:

$$\sigma = \frac{P}{A} \tag{4}$$

where:

 $\sigma$  = the engineering stress, MPa,

P = the applied, uniaxial tensile load, N, and

A =the original cross-sectional area, mm<sup>2</sup>.

Calculate the cross-sectional area, A, as:

$$A = w b \tag{5}$$

where:

w and b = the average width and average thickness of the gage section, respectively, in units of mm as detailed in 9.1.

10.3 Engineering Strain—Calculate the engineering strain as:

$$\epsilon = \frac{(I - I_o)}{I_o} \tag{6}$$

where:

 $\epsilon$  = the engineering strain,

I = the extensometer gage length at any time, and

 $I_0$  = the original gage length of the extensometer, mm.

10.3.1 In some cases the initial portion of the stress versus strain  $(\sigma - \epsilon)$  curve shows a non-linear region or 'toe' followed by a linear region as shown in Fig. 17 (c). This toe may be an artifact of the tensile test and may not represent a property of the material. The  $\sigma - \epsilon$  curve can be corrected for this toe by extending the linear region of the curve to the zero-stress point on the strain axis as shown in Fig. 17 (c). The intersection of this extension with the strain axis is the toe correction which is subtracted from all values of strain greater than the toe correction strain. The resulting  $\sigma - \epsilon$  curve is used for all subsequent calculations. Report the original stress-strain curve with the non linear toe region in uncorrected as well as corrected form.

10.4 Tensile Strength—Calculate the tensile strength as:

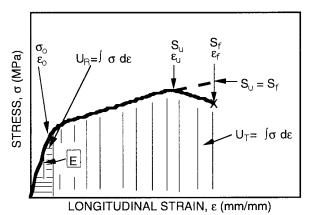
$$S_u = \frac{P_{max}}{A} \tag{7}$$

where:

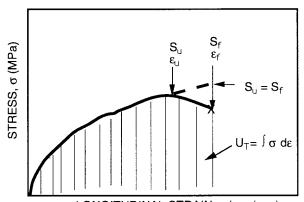
 $S_{\mu}$  = the tensile strength, MPa, and

 $P_{max}$  = the maximum load, N.

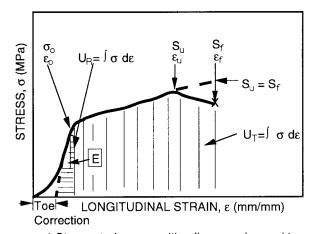
10.5 Strain at Tensile Strength—Determine strain at tensile



a) Stress-strain curve with a linear region



LONGITUDINAL STRAIN,  $\epsilon$  (mm/mm) b) Non linear stress-strain curve



c) Stress-strain curve with a linear region and toe FIG. 17 Schematic Diagrams of Stress-Strain Curves for CFCCs

strength,  $\epsilon_u$ , as the strain corresponding to the tensile strength measured during the test.

10.6 Fracture Strength—Calculate the fracture strength as:

$$S_f = \frac{P_{break}}{A} \tag{8}$$

where:

 $S_f$  = the tensile strength, MPa, and

 $P_{break}^{\prime}$  = the breaking load when the test specimen separates into two or more pieces, N.

In some instances as shown by the dashed line in Fig. 17(a), (b), (c),  $S_u = S_f$ 

10.7 Strain at Fracture Strength—Determine strain at fracture strength,  $\epsilon_f$ , as the engineering strain corresponding to the fracture strength measured during the test. In some instances as shown by the dashed line in Fig. 17(a), (b), (c),  $\epsilon_u = \epsilon_f$ .

10.8 *Modulus of Elasticity*—Calculate the modulus of elasticity as follows:

$$E = \frac{\Delta \sigma}{\Delta \epsilon} \tag{9}$$

where:

E = the modulus of elasticity, and

the slope of the  $\sigma - \epsilon$  curve within the linear region as shown in Fig. 17(*a*) and Fig. 17(*c*). The modulus of elasticity may not be defined for materials that exhibit entirely non-linear  $\sigma - \epsilon$  curves as shown in Fig. 17(*b*).

10.9 Proportional Limit Stress—Determine the proportional limit stress,  $\sigma_o$ , by one of the following methods (see Fig. 18). By its definition, the proportional limit stress,  $\sigma_o$ , may not be defined for materials which exhibit entirely non-linear  $\sigma - \epsilon$  curves as shown in Fig. 17(b).

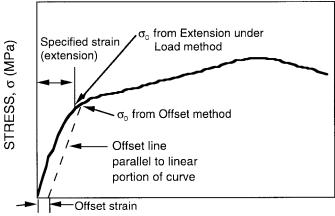
10.9.1 Offset Method—Determine  $\sigma_o$  by generating a line running parallel to the same part of the linear part of the  $\sigma - \epsilon$  curve used to determine the modulus of elasticity in 10.8. The line so generated should be at a strain offset of 0.0005 mm/mm. The proportional limit stress is the stress level at which the offset line intersects the  $\sigma - \epsilon$  curve.

10.9.2 Extension Under Load Method—Determine  $\sigma_o$  by noting the stress on the  $\sigma - \epsilon$  curve that corresponds to a specified strain. The specified strain may or may not be in the linear region of the  $\sigma - \epsilon$ , but the specified strain at which  $\sigma_o$  is determined must be constant and reported for all tests in a set.

10.10 Strain at Proportional Limit Stress—Determine strain at proportional limit stress,  $\epsilon_o$ , as the strain corresponding to proportional limit stress determined for the test.

10.11 Modulus of Resilience—Calculate the modulus of resilience as the area under the linear part of the  $\sigma-\varepsilon$  curve or alternatively estimated as:

$$U_R = \int_0^{\epsilon_o} \sigma \, d\epsilon \approx \frac{1}{2} \, \sigma_o \, \epsilon_o \tag{10}$$



LONGITUDINAL STRAIN, ε (mm/mm)

FIG. 18 Schematic Diagram of Methods for Determining Proportional Limit Stress

where  $U_R$  = the modulus of resilience in J/m<sup>3</sup> and  $\sigma_o$  and  $\epsilon_o$  as used in Eq 10 have units of Pa (that is, N/m<sup>2</sup>) and mm/mm, respectively.

10.12 *Modulus of Toughness*—Calculate the modulus of toughness as the area under the entire  $\sigma - \epsilon$  curve or alternatively estimated as:

$$U_T = \int_{o}^{\epsilon_f} \sigma \, d\epsilon \approx \frac{\sigma_o + S_u}{2} \, \epsilon_f \tag{11}$$

where  $U_T$  = the modulus of toughness in J/m³,  $\sigma_o$  and  $S_u$  as used in Eq 11 have units of Pa (that is, N/m²) and  $\epsilon_o$  has units of mm/mm.  $U_T$  can be estimated as follows for materials for which  $\sigma_o$  is not calculated and that have a  $\sigma - \epsilon$  curve that can be assumed to be a parabola.

$$U_T = \int_{\sigma}^{\epsilon_f} \sigma \, d\epsilon \approx \frac{2}{3} \, S_u \, \epsilon_f \tag{12}$$

10.13 Mean, Standard Deviation, and Coefficient of Variation—For each series of tests calculate the mean, standard deviation, and coefficient of variation for each measured value as follows:

$$Mean = X = \frac{\sum_{i=1}^{n} X_i}{n}$$
 (13)

Standard deviation = s.d. = 
$$\sqrt{\frac{\sqrt{\sum\limits_{i=1}^{n}(X_{i}-\bar{X})^{2}}}{n-1}}$$
 (14)

Coefficient of variation = 
$$V = \frac{100 (s.d.)}{X}$$
 (15)

where:

 $X_i$  = the measured values and n is the number of valid tests.

#### 11. Report

- 11.1 *Test Set*—Include in the report the following information for the test set. Note any significant deviations from the procedures and requirements of this test method.
  - 11.1.1 Date and Location of Testing:
- 11.1.2 Tensile test specimen geometry used (include engineering drawing). For end-tabbed specimens include a drawing of the tab and specify the tab material and the adhesive used.
- 11.1.3 Type and configuration of the test machine (include drawing or sketch if necessary). If a commercial test machine was used, the manufacturer and model number are sufficient for describing the test machine. Good laboratory practice also dictates recording the serial numbers of the test equipment, if available.
- 11.1.4 Type, configuration, and resolution of strain measurement equipment used (include drawing or sketch if necessary). If a commercial extensometer or strain gages were used, the manufacturer and model number are sufficient for describing the strain measurement equipment. Good laboratory practice also dictates recording the serial numbers of the test equipment, if available.
- 11.1.5 Type, configuration, and surface finish of grip interface used (include drawing or sketch if necessary). If a commercial grip interface was used, the manufacturer and model number are sufficient for describing the grip interface. Good laboratory practice also dictates recording the serial

numbers of the test equipment, if available.

- 11.1.6 Type and configuration of load-train couplers (include drawing or sketch if necessary). If a commercial load-train coupler was used, the manufacturer and model number are sufficient for describing the coupler. Good laboratory practice also dictates recording the serial numbers of the test equipment, if available.
- 11.1.7 Type and configuration of heating system (include drawing or sketch if necessary). If a commercial heating system was used, the manufacturer and model number are sufficient for describing the heating system. Good laboratory practice also dictates recording the serial numbers of the test equipment, if available.
- 11.1.8 Type and configuration of temperature measurement system (include drawing or sketch if necessary). If a commercial temperature measurement system was used, the manufacturer and model number are sufficient for describing the system. However, report the most recent calibration information. Good laboratory practice also dictates recording the serial numbers of the test equipment, if available.
- 11.1.9 Number (n) of specimens tested validly (for example, fracture in the gage section). In addition, report the total number of specimens tested ( $n_{\rm T}$ ) to provide an indication of the expected success rate of the particular specimen geometry and test apparatus.
- 11.1.10 Where feasible and possible, all relevant material data including vintage or billet identification. As a minimum, report the date the material was manufactured.
- 11.1.10.1 For commercial materials, where feasible and possible, report the commercial designation. As a minimum include a short description of reinforcement (type, layup, etc.), fiber volume fraction, and bulk density.
- 11.1.10.2 For non-commercial materials, where feasible and possible, report the major constituents and proportions as well as the primary processing routes. Also report fiber volume fraction, matrix porosity, and bulk density. Describe the reinforcement type, properties and reinforcement architecture to include fiber properties (composition, diameter, source, lot number, and any measured/specified properties), interface coatings (composition, thickness, morphology, source, and method of manufacture) and the reinforcement architecture (yarn type/count, thread count, weave, ply count, fiber areal weight, fiber fraction, stacking sequence, ply orientations, etc.).
- 11.1.11 Description of the method of specimen preparation including all stages of machining.
- 11.1.12 Where feasible and possible, heat treatments, coatings, or pre-test exposures, if any were applied either to the as-processed material or to the as-fabricated specimen.
- 11.1.13 Test environment including relative humidity (Test Method E 337), ambient temperature, and atmosphere (for example, ambient air, dry nitrogen, silicone oil, etc.), partial pressure (or percentage) of oxygen (if known), average elevated temperature, and average hold time at elevated temperature.
- 11.1.14 Test mode (load, displacement, or strain control) and actual test rate (load rate, displacement rate, or strain rate). Report calculated strain rate, if appropriate, in units of  $s^{-1}$ .
  - 11.1.15 Percent bending and corresponding average strain



in the specimen recorded during the verification as measured at the beginning and end of the test series.

- 11.1.16 Mean, standard deviation, and coefficient of variation of the following measured properties for each test series:
  - 11.1.16.1 Tensile strength,  $S_{\rm u}$ ,
  - 11.1.16.2 Strain at tensile strength,  $\epsilon_{uv}$
  - 11.1.16.3 Fracture strength,  $S_{t}$
  - 11.1.16.4 Strain at fracture strength,  $\epsilon_{f}$
  - 11.1.16.5 Modulus of elasticity, E (if applicable),
- 11.1.16.6 Proportional limit stress,  $\sigma_{o}$ (if applicable) and method of determination,
- 11.1.16.7 Strain at proportional limit stress,  $\epsilon_o$  (if applicable),
  - 11.1.16.8 Modulus of resilience,  $U_R$  (if applicable), and
  - 11.1.16.9 Modulus of toughness,  $U_T$  (if applicable).
- 11.2 *Individual Specimens*—Report the following information for each specimen tested. Note and report any significant deviations from the procedures and requirements of this test method:
- 11.2.1 Temperature of test, K, time to attain test temperature, time at temperature prior to testing, and test environment,
- 11.2.2 Pertinent overall specimen dimensions, if measured, such as total length, length of gage section, gripped section dimensions, etc. in mm.
- 11.2.3 Average surface roughness of the gage section,  $\mu m$ , if measured, and the direction of measurement,
- 11.2.4 Average cross-sectional dimensions, if measured, or cross-sectional dimensions at the plane of fracture, mm,

- 11.2.5 Plot of the entire stress-strain curve,
- 11.2.6 Tensile strength,  $S_{uv}$
- 11.2.7 Strain at tensile strength,  $\epsilon_u$ ,
- 11.2.8 Fracture strength,  $S_{t}$
- 11.2.9 Strain at fracture strength,  $\epsilon_{f}$
- 11.2.10 Modulus of elasticity, E (if applicable),
- 11.2.11 Proportional limit stress,  $\sigma_o$  (if applicable) and method of determination,
  - 11.2.12 Strain at proportional limit stress,  $\epsilon_o$  (if applicable),
  - 11.2.13 Modulus of resilience,  $U_R$  (if applicable),
  - 11.2.14 Modulus of toughness,  $U_T$  (if applicable),
- 11.2.15 Fracture location relative to the gage section midpoint, mm (+ is toward the top of the specimen as marked and—is toward the bottom of the specimen as marked with 0 being the gage section midpoint), and
- 11.2.16 Appearance of specimen after fracture as suggested in 9.5.

#### 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 of this test method.

### 13. Keywords

13.1 ceramic matrix composite; CFCC; continuous fiber composite; elevated temperature; tensile test

# APPENDIX

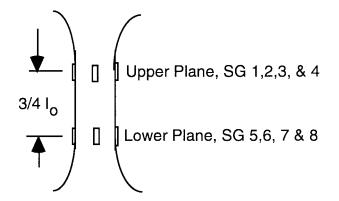
(Nonmandatory Information)

### X1. VERIFICATION OF LOAD TRAIN ALIGNMENT AT ROOM TEMPERATURE

- X1.1 Purpose of Verification—The purpose of this verification procedure is to demonstrate that the grip interface and load-train couplers can be used by the test operator in such a way as to consistently meet the limit on percent bending as specified in 6.5. Thus, in conducting this verification procedure use no more care in setup than that used in the routine testing of the actual tensile specimen. Measure the bending under tensile load using verification (or actual) specimens of the same design as that to be used for the tensile tests. For the verification purposes, apply strain gages as shown in Fig. X1.1. Conduct verification measurements: at the beginning and end of a series of tests with a measurement at the midpoint of the series recommended, whenever the grip interfaces and loadtrain couplers are installed on a different test machine, whenever a different operator is conducting a series of tests, and whenever damage or misalignment is suspected. Since the verification specimen uses adhesively bonded strain gages, conduct the verification procedure at room temperature mindful that this implies that the load-train alignment will remain constant at elevated temperatures.
- X1.2 Verification Specimen—Machine the specimen used for verification very carefully with attention to all tolerances

and concentricity requirements. Ideally, the verification specimen should 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, use an alternate material (isotropic, homogeneous, continuous) with similar elastic modulus, elastic strain capability, and hardness to the test material. Carefully inspect the specimen with an optical comparator before strain gages are attached to ensure that these dimensional requirements are met. Exercise care in handling and using the verification specimen since, after the strain gages are applied, it will no longer be possible to meaningfully inspect the specimen.

X1.2.1 For simplicity, mount a minimum of eight foil resistance strain gages on the verification specimen as shown in Fig. X1.1. Separate the strain gage planes by  $\sim \frac{3}{4} l_o$  where  $l_o$  is the length of the reduced or designated gage section. Mount four strain gages, equally spaced (90° apart) around the circumference of the gage section (that is, one strain gage on each face), at each of two planes at either end of the gage section. Ensure that the longitudinal centers of all strain gages on the same plane are within 0.5 mm of the same longitudinal



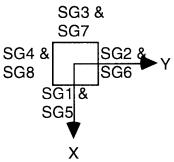


FIG. X1.1 Illustration of Strain Gage Placement on Gage Section Planes and Strain Gage Numbering

distance along the specimen axis. These planes shall be symmetrically located about the longitudinal midpoint of the gage section. Employ suitable strain recording equipment.

Note X1.1—Take care to select strain gage planes that are symmetrical about the longitudinal midpoint of the gage section. Avoid placing the strain gages closer than one strain gage length from geometrical features such as the transition radius from the gage section. Such placement can cause strain concentrations and inaccurate measures of the strain in the uniform gage section. Strain gages on dummy specimens composed of isotropic homogeneous materials should be as narrow as possible to minimize strain averaging. Strain gages having active widths of 0.25 to 0.5 mm and active lengths of 1.0 to 2.5 mm are commercially available and are suitable for this purpose. Otherwise, sizes of strain gages on test specimens composed of CFCC materials must be chosen accordingly. Unless it can be shown that strain gage readings are not unduly influenced by localized strain events such as fiber crossovers, strain gages should not be less than 9 to 12 mm in length for the strain-measurement direction and not less than 6 mm in width for the direction normal to strain measurement. Larger strain gages than those recommended here may be required for fabric reinforcements to average the localized strain effects of the fiber crossovers. Choose the strain gages, surface preparation, and bonding agents so as to provide adequate performance on the subject materials. Many CFCCs may exhibit high degrees of porosity and surface roughness and therefore require surface preparation including surface filling before the strain gages can be applied.

X1.3 Verification Procedure—Procedures for verifying alignment are described in detail in Practice E 1012. However, salient points and equations for square and circular cross-sections as currently contained in Practice E 1012 are described here for emphasis. Consult Practice E 1012 for specific details for rectangular cross-sections, especially when the thickness is too thin to strain gage all four sides. The following paragraphs are not intended to replace Practice E 1012, but

rather are intended to elucidate those aspects which are directly applicable to this particular test method.

X1.3.1 Mount the top of the specimen in the grip interface.

X1.3.2 Connect the lead wires of the strain gages to the conditioning equipment and allow the strain gages to equilibrate under power for at least 30 min prior to conducting the verification tests. This will minimize drift during actual conduct of the verifications.

X1.3.3 Zero the strain gages before mounting the bottom of the specimen in the grip interface. This will allow any bending due to the grips to be recorded.

X1.3.4 Mount the bottom of the specimen in the grip interface.

X1.3.5 Apply a sufficient load to the specimen to achieve a mean strain equal to either one-half the anticipated strain at the onset of the cumulative fracture process (for example, matrix cracking stress) in the test material or a strain of 0.0005 (that is, 500 micro strain) whichever is greater. It is desirable to record the strain (and hence percent bending) as functions of the applied load to monitor any self alignment of the load train.

X1.3.6 Calculate percent bending as follows for square cross sections referring to Fig. X1.1 for the strain gage numbers. Calculate percent bending at the upper plane of the gage section as:

$$PB_{upper} = \frac{\epsilon_b}{\epsilon} 100 \tag{X1.1}$$

$$\epsilon_b = \left[ \left( \frac{\epsilon_1 - \epsilon_3}{2} \right)^2 + \left( \frac{\epsilon_2 - \epsilon_4}{2} \right)^2 \right]^{\frac{1}{2}}$$
 (X1.2)

$$\epsilon_o = \frac{\epsilon_1 + \epsilon_2 + \epsilon_3 + \epsilon_4}{4} \tag{X1.3}$$

where  $\epsilon_1$ ,  $\epsilon_2$ ,  $\epsilon_3$ , and  $\epsilon_4$  are strain readings for strain gages located at the upper plane of the gage section. Strain gage readings are in units of strain, m/m, and compressive strains are negative.

X1.3.7 Calculate percent bending at the lower plane of the gage section for square cross sections referring to Fig. X1.1 for the strain gage numbers as follows:

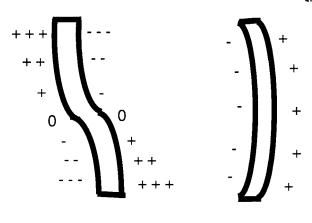
$$PB_{lower} = \frac{\epsilon_b}{\epsilon} 100 \tag{X1.4}$$

$$\epsilon_b = \left[ \left( \frac{\epsilon_5 - \epsilon_7}{2} \right)^2 + \left( \frac{\epsilon_6 - \epsilon_8}{2} \right)^2 \right]^{\frac{1}{2}}$$
 (X1.5)

$$\epsilon_o = \frac{\epsilon_5 + \epsilon_6 + \epsilon_7 + \epsilon_8}{4}$$
 (X1.6)

where  $\epsilon_5$ ,  $\epsilon_6$ ,  $\epsilon_7$ , and  $\epsilon_8$  are strain readings for strain gages located at the lower plane of the gage section. Strain gage readings are in units of strain, m/m, and compressive strains are negative.

X1.3.8 For uniform bending across the gage section with the specimen assuming a C-shape,  $PB_{upper} \approx PB_{lower}$ . C-shape bending reflects angular misalignment of the grips. For non-uniform bending across the gage section with the specimen assuming a S-shape,  $PB_{upper}$  may or may not be equal to  $PB_{lower}$ . S-shape bending reflects eccentric misalignment of the grip centerlines. These general tendencies are shown in Fig. X1.2. Combinations of C and S shapes may exist. In these cases the S-shape should first be eliminated by adjusting the



S-shape C-shape
FIG. X1.2 S-Shape and C-Shape Bending of Tensile Specimen

eccentricity of the grips such that the longitudinally aligned strain gages indicate approximately the same values (for example,  $\epsilon_1 \approx \epsilon_5$ ,  $\epsilon_2 \approx \epsilon_6$ , etc.). More detailed discussions regarding bending and alignment are contained in (15).

X1.3.9 Check the effect of the specimen warpage by rotating the specimen 180° about its longitudinal axis and performing the bending checks again. If similar results are obtained at each rotation then the degree of alignment can be considered representative of the load train and not indicative of the specimen. If load-train alignment is within the specifications of 6.5, record the maximum percent bending and conduct the tensile tests. If the load-train alignment is outside the specifications of 6.5 then realign or readjust the load train according to the specific procedures unique to the individual testing setup. Repeat this verification procedure to confirm the achieved alignment.

#### REFERENCES

- (1) Jenkins, M.G., "Long-Term Testing of Advanced Ceramics: Concerns, Insights, and Recommendations," *Paper 95-GT-237*, American Society of Mechanical Engineers, New York, 1995.
- (2) Hartman, G.A., Zawada, L.P., and Russ, S.M., "Techniques for Elevated-Temperature Testing of Advanced Ceramic Composite Materials," *Proceedings of the Fifth Annual Hostile Environment and High Temperature Measurement Conference*, Society for Experimental Mechanics, Bethel, CT, 1988, pp. 31–38.
- (3) Lewis, D, III, "Tensile Testing of Ceramics and Ceramic-Matrix Composites," *Tensile Testing*, ASM International, Materials Park, OH, 1992, pp. 147–181.
- (4) Holmes, J.W., "A Technique for Elevated-Temperature Creep and Tensile Fatigue Testing of Fiber-Reinforced Ceramics," J. Compos. Mater., 26, [6], 1992, pp. 915–932.
- (5) Barnett, T.R., and Starrett, H.S., "Room and Elevated-Temperature Mechanical and Thermal Properties of Corning Nicalon/CAS" WRDC-TR-90-4131, Wright Research and Development Center, Wright Patterson Air Force Base, OH, 1992.
- (6) Bartlett, M.L., "Expectations and Elevated-Temperature Testing of Advanced Materials Used for the Development of NASP," Proceedings of International Symposium on Ultra-High Temperature Materials, Ube, Japan, 1991.
- (7) Amaral, J.E., and Pollock, C.N., "Machine Design Requirements for Uniaxial Testing of Ceramics Materials," *Mechanical Testing of Engineering Ceramics at High Temperatures*, B.F. Dyson, R.D. Lohr, and R. Morrell, eds., 1989, pp. 51–68.
- (8) Mosiman, L.G., Wallenfelt, T.L., and Larsen, C.G., "Tension/

- Compression Grips for Monolithic Ceramics and Ceramic Matrix Composites," *Ceramic Engineering and Science Proceeding*, 12 (7–8), 1991.
- (9) Liu, K.C. and Brinkman, C.R., "Tensile Cyclic Fatigue of Structural Ceramics," Proceedings of the 23rd Automotive Technology Development Contractors' Coordination Meeting, P-165, SAE Warrendale, PA, 1986, pp. 279–284.
- (10) Mejia, L.C., "High Temperature Tensile Testing of Advanced Ceramics," Ceramic Engineering and Science Proceedings, 10 (7–8), 1989, pp. 668–681.
- (11) Jenkins, M.G., Ferber, M.K., Martin, R.L., Jenkins, V.T., Tennery, V.J., "Study and Analysis of the Stress State in a Ceramic, Button-Head, Tensile Specimen," Oak Ridge National Laboratory Technical Memorandum, ORNL/TM 11767, September 1991.
- (12) Manual on the Use of Thermocouples in Temperature Measurement, ASTM MNL 12, ASTM, 1993.
- (13) Worthem, D. W., "Flat Tensile Specimen Design for Advanced Composites," NASA CR-185261, November 1990.
- (14) Shuler, S. F., Holmes, J. W., Research Memorandum No. 102, "Influence of Loading Rate on the Monotonic Tensile Behavior of Fiber Reinforced Ceramics", available through: Ceramic Composites Research Laboratory, Dept. of Mechanical Engineering and Applied Mechanics, 1065 GGBL, The University of Michigan, Ann Arbor, MI 48109-2125, September 1990.
- (15) Larsen, C.G., Ceramics Tensile Grip," ASTM STP 1080, J.M. Kennedy, H.H. Moeller, and W.W. Johnson, eds., ASTM, 1990, pp. 235–246.

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