



Designation: **C1468 – 06 C1468 – 13**

## Standard Test Method for Transthickness Tensile Strength of Continuous Fiber- Reinforced Advanced Ceramics at Ambient Temperature<sup>1</sup>

This standard is issued under the fixed designation C1468; 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—Scope\*

1.1 This test method covers the determination of transthickness tensile strength ( $S'_T$ ) under monotonic uniaxial forcing of continuous fiber-reinforced ceramics (CFCC) at ambient temperature. This test method addresses, but is not restricted to, various suggested test specimen geometries, test fixtures, data collection and reporting procedure. In general, round or square test specimens are tensile tested in the direction normal to the thickness by bonding appropriate hardware to the samples and performing the test. For a Cartesian coordinate system, the  $x$ -axis and the  $y$ -axis are in the plane of the test specimen. The transthickness direction is normal to the plane and is labeled the  $z$ -axis for this test method. For CFCCs, the plane of the test specimen normally contains the larger of the three dimensions and is parallel to the fiber layers for uni-directional, bi-directional, and woven composites. Note that transthickness tensile strength as used in this test method refers to the tensile strength obtained under monotonic uniaxial forcing where monotonic refers to a continuous nonstop test rate with no reversals from test initiation to final fracture.

1.2 This test method is intended primarily for use with all advanced ceramic matrix composites with continuous fiber reinforcement: unidirectional (1-D), bidirectional (2-D), woven, and tridirectional (3-D). In addition, this test method also may be used with glass (amorphous) matrix composites with 1-D, 2-D, and 3-D continuous fiber reinforcement. This test method does not address directly discontinuous fiber-reinforced, whisker-reinforced or particulate-reinforced ceramics, although the test methods detailed here may be equally applicable to these composites. It should be noted that 3-D architectures with a high volume fraction of fibers in the “ $z$ ” direction may be difficult to test successfully.

1.3 Values are in accordance with the International System of Units (SI) and **IEEE/ASTM SI 10**.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Additional recommendations are provided in **6.7** and Section **7**.

### 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

**C1145** Terminology of Advanced Ceramics

**C1239** Practice for Reporting Uniaxial Strength Data and Estimating Weibull Distribution Parameters for Advanced Ceramics

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

**C1468** Test Method for Transthickness Tensile Strength of Continuous Fiber-Reinforced Advanced Ceramics at Ambient Temperature

**D3878** Terminology for Composite Materials

**E4** Practices for Force Verification of Testing Machines

**E6** Terminology Relating to Methods of Mechanical Testing

**E177** Practice for Use of the Terms Precision and Bias in ASTM Test Methods

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

**E691** Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

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

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

\*A Summary of Changes section appears at the end of this standard

IEEE/ASTM SI 10 American National Standard for Use of the International System of Units (SI): The Modern Metric System.  
 E1012 Practice for Verification of Testing Frame and Specimen Alignment Under Tensile and Compressive Axial Force Application

### 3. Terminology

#### 3.1 Definitions:

3.1.1 The definitions of terms relating to tensile testing appearing in Terminology E6 apply to the terms used in this test method. The definitions of terms relating to advanced ceramics appearing in Terminology C1145 apply to the terms used in this test method. The definitions of terms relating to fiber-reinforced composites appearing in Terminology D3878 apply to the terms used in this test method. Pertinent definitions as listed in Practice E1012, Terminology C1145, Terminology D3878, and Terminology E6 are shown in the following with the appropriate source given in brackets. Terms used in conjunction with this test method are defined as follows:

3.1.2 *advanced ceramic, n*—a highly-engineered, high-performance predominately nonmetallic, inorganic, ceramic material having specific functional attributes. [C1145]

3.1.3 *bending strain, n*—the difference between the strain at the surface and the axial strain. [E1012]

3.1.4 *breaking force, n*—the force at which fracture occurs,  $P_{max}$ , is the breaking force in units of N. [E6]

3.1.5 *ceramic matrix composite (CMC), 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. [C1145]

3.1.6 *continuous fiber-reinforced ceramic matrix composite (CFCC), n*—a ceramic matrix composite in which the reinforcing phases consists of continuous filaments, fibers, yarn, or knitted or woven fabrics. [C1145]

3.1.7 *gage length, n*—the original length [ $L_{GL}$ ] of that portion of the test specimen over which strain or change of length is determined. [E6]

3.1.8 *modulus of elasticity, n*—the ratio of stress to corresponding strain below the proportional limit. [E6]

3.1.9 *percent bending, n*—the bending strain times 100 divided by the axial strain. [E1012]

3.1.10 *tensile strength, n*—the maximum tensile stress, which a material is capable of sustaining. Tensile strength is calculated from the maximum force during a tension test carried to rupture and the original cross-sectional area of the test specimen. [E6]

#### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *transthickness, n*—the direction parallel to the thickness, that is, out-of-plane dimension, as identified in 1.1, and also typically normal to the plies for 1-D, 2-D laminate, and woven cloth. For 3-D laminates this direction is typically taken to be normal to the thickness and associated with the “z” direction.

3.2.2 *fixturing, n*—fixturing is referred to as the device(s) bonded to the test specimen. It is this device(s) that is actually gripped or pinned to the force train. The fixturing transmits the applied force to the test specimen.

### 4. Significance and Use

4.1 This test method may be used for material development, material comparison, quality assurance, characterization, and design data generation.

4.2 Continuous fiber-reinforced ceramic matrix composites generally are characterized by fine grain sized (<50  $\mu\text{m}$ ) glass or ceramic matrices and ceramic fiber reinforcements. CFCCs are candidate materials for high-temperature structural applications requiring high degrees of corrosion and oxidation resistance, wear resistance, and inherent damage tolerance, that is, toughness. In addition, continuous fiber-reinforced glass (amorphous) matrix composites are candidate materials for similar but possibly less-demanding applications. Although shear test methods are used to evaluate shear interlaminar strength ( $\tau_{ZX}$ ,  $\tau_{ZY}$ ) in advanced ceramics, there is significant difficulty in test specimen machining and testing. Improperly prepared notches can produce nonuniform stress distribution in the shear test specimens and can lead to ambiguity of interpretation of strength results. In addition, these shear test specimens also rarely produce a gage section that is in a state of pure shear. Uniaxially-forced transthickness tensile strength tests measure the tensile interlaminar strength ( $S_T^T$ ), avoid the complications listed above, and provide information on mechanical behavior and strength for a uniformly stressed material. The ultimate strength value measured is not a direct measure of the matrix strength, but a combination of the strength of the matrix and the level of bonding between the fiber, fiber/matrix interphase, and the matrix.

4.3 CFCCs tested in a transthickness tensile test may fail from a single dominant flaw or from a cumulative damage process; therefore, the volume of material subjected to a uniform tensile stress for a single uniaxially-forceed transthickness tensile test may be a significant factor in determining the ultimate strength of CFCCs. The probabilistic nature of the strength distributions of the brittle matrices of CFCCs requires a sufficient number of test specimens at each testing condition for statistical analysis and design, with guidelines for test specimen size and sufficient numbers provided in this test method. Studies to determine the exact influence

of test specimen volume on strength distributions for CFCCs have not been completed. It should be noted that strengths obtained using other recommended test specimens with different volumes and areas may vary due to these volume differences.

4.4 The results of transthickness tensile tests of test 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.

4.5 For quality control purposes, results derived from standardized transthickness tensile test specimens may be considered indicative of the response of the material from which they were taken for given primary processing conditions and post-processing heat treatments.

4.6 The strength of CFCCs is dependent on their inherent resistance to fracture, the presence of flaws, or damage accumulation processes, or a combination thereof. Analysis of fracture surfaces and fractography, though beyond the scope of this test method, is highly 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 strength. In particular, the behavior of materials susceptible to slow crack growth fracture will be strongly influenced by test environment and testing rate. Testing to evaluate the maximum strength potential of a material should be conducted in inert environments or at sufficiently rapid testing rates, or both, so as to minimize slow crack growth effects. Conversely, testing can be conducted in environments and testing modes and rates representative of service conditions to evaluate material performance under use conditions. When testing is conducted in uncontrolled ambient air with the intent of evaluating maximum strength potential, relative humidity, and temperature must be monitored and reported. Testing at humidity levels >65 % RH is not recommended and any deviations from this recommendation must be reported.

5.2 Surface and edge preparation of test specimens, although normally not considered a major concern in CFCCs, can introduce fabrication flaws which may have pronounced effects on the measured transthickness strength **(1)**.<sup>3</sup> Machining damage introduced during test specimen preparation can be either a random interfering factor in the determination of strength of pristine material, that is, increased frequency of surface-initiated fractures compared to volume-initiated fractures, or an inherent part of the strength characteristics. Universal or standardized test methods of surface and edge preparation do not exist. It should be understood that final machining steps may, or may not, negate machining damage introduced during the initial machining; thus, test specimen fabrication history may play an important role in the measured strength distributions and should be reported. In addition, the nature of fabrication used for certain composites, for example, chemical vapor infiltration or hot pressing, may require the testing of test specimens in the as-processed condition.

5.3 Bending in uniaxial transthickness tensile tests can cause or promote nonuniform stress distributions with maximum stresses occurring at the test specimen edge leading to nonrepresentative fractures. Similarly, fracture from edge flaws may be accentuated or suppressed by the presence of the nonuniform stresses caused by bending.

NOTE 1—Finite element calculations were performed for the square cross section test specimen for the forcing conditions and test specimen thickness investigated in reference **(1)**. Stress levels along the four corner edges were found to be lower than the interior, except for the corners at the bond lines where the stress was slightly higher than the interior. Stress levels along the sides and interior of the test specimen were found to be uniform.

## 6. Apparatus

6.1 *Testing Machines*—Machines used for transthickness tensile testing shall conform to the requirements of Practice **E4**. The forces used in determining tensile strength shall be accurate within  $\pm 1\%$  at any force within the selected force range of the testing machine as defined in Practice **E4**. A schematic showing pertinent features of the transthickness tensile testing apparatus for two possible forcing configurations is shown in **Figs. 1 and 2**.

6.1.1 Values for transthickness tensile strength can range a great deal for different types of CFCC. Therefore, it is helpful to know an expected strength value in order to properly select a force range. Approximate transthickness tensile strength values **(1)** for several CFCCs are as follows: porous oxide/oxide composites range from 2–10 MPa, hot pressed fully dense SiC/MAS-5 glass-ceramic composites range from 14–27 MPa, Polymer Infiltrated and Pyrolyzed (PIP) SiC/SiNC range from 15–32 MPa, and hot pressed SCS-6/Si<sub>3</sub>N<sub>4</sub> range from 30–43 MPa.

6.1.2 For any testing apparatus, the force train will need to be aligned for angularity and concentricity. Alignment of the testing system will need to be measured and is detailed in A1.1 of Test Method **C1275**.

### 6.2 Gripping Devices:

6.2.1 *General*—Various types of gripping devices may be used to transmit the force applied by the testing machine to the test fixtures and into the test specimens. The brittle nature of the matrices of CFCCs requires accurate alignment. Bending moments can produce stresses leading to premature crack initiation and fracture of the test specimen. Gripping devices can be classified generally as those employing active and those employing passive grip interfaces as discussed in the following sections. Several additional gripping techniques are discussed in Test Method **C1275**.

<sup>3</sup> The boldface numbers in parentheses refers to the list of references at the end of this standard.

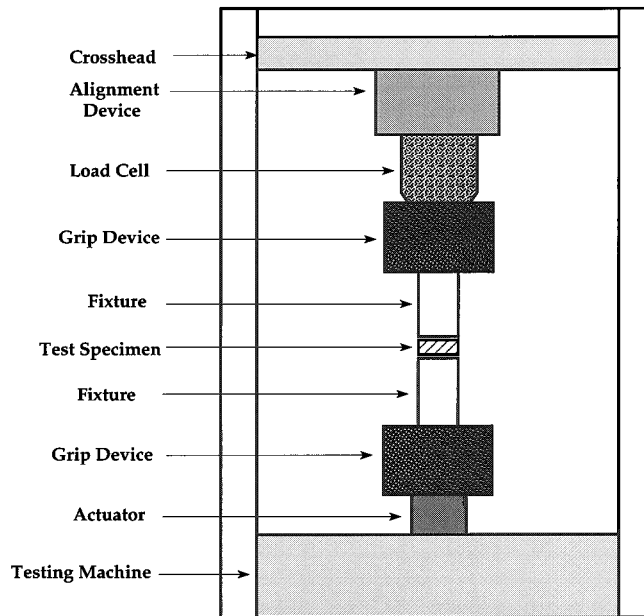


FIG. 1 Schematic Diagram of One Possible Apparatus for Conducting a Uniaxially-Forced Transthickness Tensile Test

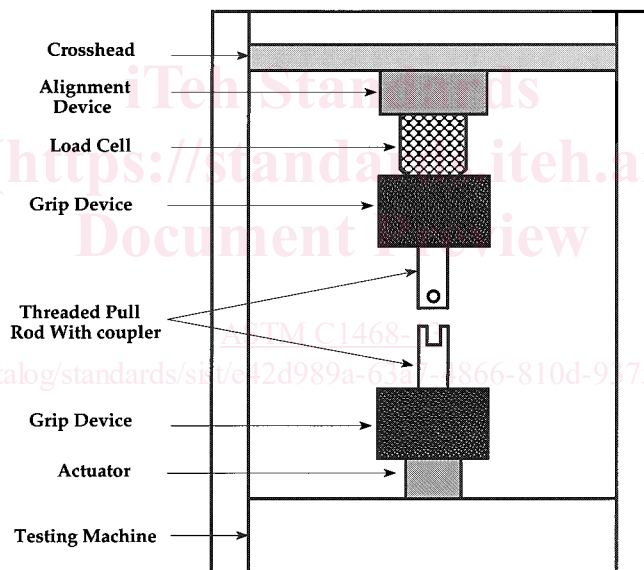


FIG. 2 Schematic Diagram of a Second Possible Apparatus for Conducting a Uniaxially-Forced Transthickness Tensile Test

6.2.1.1 *Active Grip Interfaces*—Active grip interfaces require a continuous application of a mechanical, hydraulic, or pneumatic force to transmit the force applied by the test machine to the test fixtures. Generally, these types of grip interfaces cause a force to be applied normal to the surface of the gripped section of the test fixturing. Transmission of the uniaxial force applied by the test machine then is accomplished by friction between the test fixturing and the grip faces; thus, important aspects of active grip interfaces are uniform contact between the gripped section of the test fixturing and the grip faces and constant coefficient of friction over the grip/fixture interface. In addition, for active grips, uniform application of gripping force and motion of the grips upon actuation are important factors to consider in assuring proper gripping.

(1) Face-forced grips, either by direct lateral pressure grip faces (2) or by indirect wedge-type grip faces, act as the grip interface (3). Generally, close tolerances are required for the flatness and parallelism as well as for the wedge angle of the wedge grip faces. In addition, the thickness, flatness, and parallelism of the gripped section of the fixturing shall be within similarly close tolerances to promote uniform contact at the fixture/grip interface. Tolerances will vary depending on the exact configuration.

(2) Sufficient lateral pressure should be applied to prevent slippage between the grip face and the fixturing. Grip surfaces that are scored or serrated with a pattern similar to that of a single-cut file have been found satisfactory. A fine serration appears to be the most satisfactory. The serrations should be kept clean and well defined but not overly sharp. The length [L] and width [W] of the grip faces should be equal to or greater than the respective length and width of the fixturing to be gripped.

(3) Grip inserts, called wedges, can be machined to accept flat or round fixturing. This allows for a wide range of fixturing to be utilized.

6.2.1.2 *Passive Grip Interfaces*—Passive grip interfaces transmit the force applied by the test machine through a direct mechanical link (4). Generally, these mechanical links transmit the test forces to the test specimen via geometrical features of the test fixturing. Passive grips may act through pin forcing via pins at holes in the fixturing. Generally, close tolerances of linear dimensions are required to promote uniform contact as well as to provide for noneccentric forcing. In addition, moderately close tolerances are required for center-line coincidence and diameter [ $D$ ] of the pins and holes.

6.3 *force Train Couplers:*

6.3.1 *General*—Various types of devices (force train couplers) may be used to attach the active or passive grip interface assemblies to the testing machine (1,5,6,7). The force train couplers in conjunction with the type of gripping device play major roles in the alignment of the force train, and thus, subsequent bending imposed in the test specimen. force train couplers can be classified generally as fixed and non-fixed as discussed in the following sections. Note that use of well-aligned fixed or self-aligning non-fixed couplers does not automatically guarantee low ending in the test specimen. The type and operation of grip interfaces, as well as the as-fabricated dimensions of the test specimen can add significantly to the final bending imposed in the test specimen. Additional information pertaining to couplers can be found in Test Method C1275.

6.3.1.1 Verify alignment of the testing system as a minimum at the beginning and end of a test series as detailed in A1.1 of Test Method C1275, unless the conditions for verifying alignment additional times are met. A test series is a discrete group of tests on individual test specimens conducted within a discrete period of time on a particular material configuration, test specimen geometry, test condition, or other uniquely definable qualifier, for example, a test series composed of material A comprising ten test specimens of geometry B tested at a fixed rate in force control to final fracture in ambient air. An additional verification of alignment is recommended, although not required, at the middle of the test series. Measure alignment with a dummy test specimen and the alignment verification procedures detailed in Test Method C1275. Allowable bending values are discussed in 6.4. Alignment test specimens used for verification should be equipped with a recommended eight separate longitudinal strain gages to determine bending contributions from both concentric and angular misalignment of the grip heads. The length of the alignment test specimen should be approximately the same length as the test specimen and fixturing. Use a material (isotropic, homogeneous, continuous) with similar elastic modulus and elastic strain capability to the CFCC being tested.

6.3.2 *Fixed force Train Couplers* —Fixed couplers may incorporate devices which require either a one-time, pre-test alignment adjustment of the force train, which remains constant for all subsequent tests or an in-situ, pre-test alignment of the force train which is conducted separately for each test specimen and each test. Such devices (8) usually employ angularity and concentricity adjusters to accommodate inherent force train misalignments. Fixed force trains have two translational degrees of freedom and three degrees of rotational freedom fixed. Regardless of which method is used, verify the alignment as discussed in 6.3.1.1. A schematic diagram of one possible arrangement for a fixed force train is shown in Fig. 3, and this arrangement corresponds to the

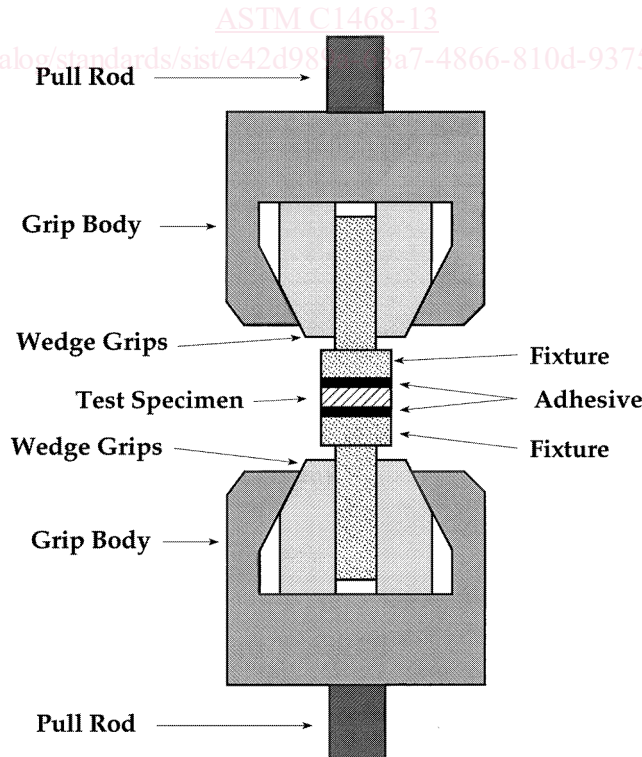


FIG. 3 Schematic Diagram of One Possible Arrangement for a Fixed-force Train

force train identified in Fig. 1.

6.3.2.1 Fixed force train couplers often are preferred for monotonic testing CFCCs. During the fracture process, the fixed coupler tends to hold the test specimen in an aligned position, and thus, provides a continuous uniform stress across the remaining ligament of the gage section. For transthickness tensile testing, however, this is not an issue, allowing for use of both methods.

6.3.2.2 The use of fixed force train coupler typically will require that the test specimens be bonded to the fixturing after the fixturing has been mounted in the test frame or grips. CFCCs in general have low transthickness tensile strength, as stated in 6.1.1, and this requirement will minimize the possibility of inducing bending when the fixturing is gripped. One drawback to mounting the test specimen in the force frame is that it will reduce productivity. There will be a waiting period as the adhesive cures. Care must be taken to insure that the test specimen does not move on the fixturing during the cure cycle of the adhesive.

6.3.3 *Nonfixed force Train Couplers*—Nonfixed couplers may incorporate devices which promote self-alignment of the force train during the movement of the crosshead or actuator. Generally, such devices rely upon freely moving linkages to eliminate applied moments as the force train components are forced. Knife edges, universal joints, hydraulic couplers or air bearings are examples (5,9,10) of such devices. Although nonfixed force train couplers are intended to be self-aligning, the operation of the couplers must be verified as discussed in 6.3.1.1. A schematic diagram of one possible arrangement for a nonfixed force train is shown in Fig. 4, and this arrangement corresponds to the force train identified in Fig. 2.

NOTE 2—The use of nonfixed force train couplers allows for many test specimens to be prepared ahead of time using an alignment device. Once the test specimens are bonded to the fixturing, they can all be tested in a very short period of time. This greatly increases throughput and minimizes machine time.

6.3.3.1 The forcing configuration shown in Fig. 4 uses universal rod ends (sometimes called ball joint rod ends) at both ends of the fixtured test specimen. The universal rods allow for a full range of angular motion and will allow for some concentricity and angularity misalignment of the grips. A photograph showing assembly of the fixturing, test specimen, and universal rod ends is shown in Fig. 5.

6.4 *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,  $m'$ , and characteristic strength,  $\sigma_0$ ) of monolithic advanced ceramics, allowable percent bending as defined in Practice E1012 should not exceed five. Conclusions arrived at in (11) for the uniaxial tension strength along one of the directions of reinforcement are also supposed to be valid for the transthickness case. Applying these conclusions for this test method (11) assumes that transthickness tensile strength fractures are due to single fracture origins in the volume of the material, all test specimens experience the same level of bending, and that Weibull modulus,  $m'$ , was constant.

6.4.1 Studies of the effect of bending on the transthickness 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

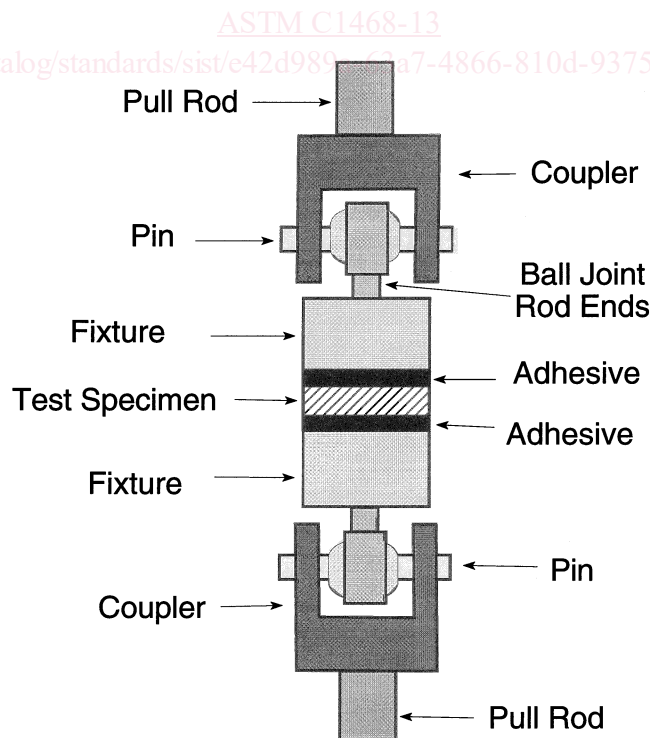
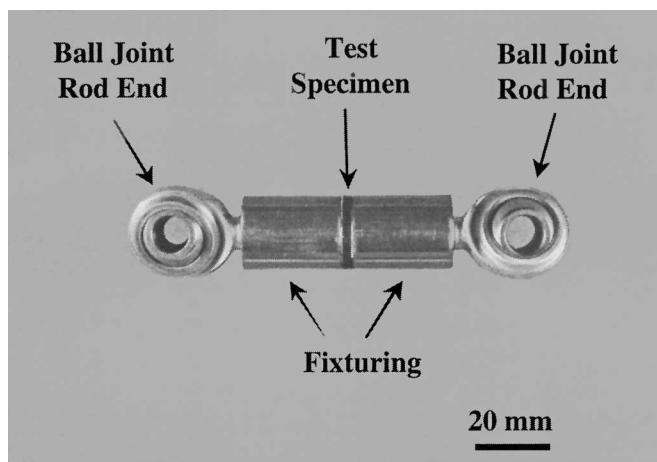


FIG. 4 Schematic Diagram of One Possible Arrangement for a Nonfixed force Train That Uses Couplers and Ball Joint Rod End Adapters



**FIG. 5 Photograph of a Transthickness Tensile Test Specimen Bonded to Fixturing, With Fixturing Assembled with Universal Rod Ends (Ball Joint Rod Ends) for Improved Alignment**

ceramics and uniaxial tensile testing of CFCCs. The recommended maximum allowable percent bending at the onset of the cumulative fracture process, for example, matrix cracking stress, for test specimens tested under this standard is five at the anticipated fracture force.

**6.5 Data Acquisition**—At the minimum, make an autographic record of maximum force; however, it is desirable to also make a record, where applicable, of applied force, cross-head displacement, strain, and time. Use either analog chart recorders or digital data acquisition systems for this purpose, although a digital record is recommended for ease of later data analysis. Recording devices shall be accurate to 1.0 % of full scale. Data acquisition rates will depend on the forcing rates used to conduct the test. A data acquisition rate of at least 20 Hz should be used, and the acquisition rate should be fast enough to capture the maximum force within 1 %.

**6.6 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, measure cross-sectional dimensions to within 0.02 mm, requiring measuring devices with accuracy of 0.01 mm.

**6.7 Adhesives**—In conducting a transthickness tensile test, an adhesive is required to bond the test specimen to the fixturing, as it is not normally possible to directly grip the test specimen. There are many types of adhesives available, and care should be taken to select an adhesive strong enough to conduct the test.

**NOTE 3**—Many adhesives contain hazardous chemicals. Manufacturers of adhesives routinely provide listings of the possible hazards associated with particular adhesives, and commonly provide Material Safety Data Sheets (MSDS) on their products. Read all safety handling requirements and follow the manufacturers recommended handling procedures. In general, always utilize protective face, eye, hand, and body gear. If the adhesive produces gases, use only in vented hoods certified for those specific gases.

**6.7.1** The strength of the adhesive can be evaluated by bonding the fixturing together without the test specimen and performing the transthickness tension test on just the adhesive. The tensile strength of the adhesive then can be determined as described in 10.3.

**6.7.2** Single-part adhesives that air cure at room temperature are the easiest to use, but generally exhibit low strength.

**6.7.3** Two-part adhesives require a bulk resin, along with a catalyst to activate curing. These adhesives demonstrate moderate strength, and often require glass beads of a specific size to produce a bond line of specific thickness for optimum bonding. Often, there is excess adhesive present when trying to insure a complete bond line, and this can pose a problem, as adhesive should not flow up or down the edges of the test specimen; therefore, care should be taken in the amount of adhesive used.

**6.7.4** Single-part adhesives that cure at an elevated-temperature are very easy to handle and generally produce very high-strength bonds. Several of these elevated temperature curing adhesives are produced in sheets that easily are cut to the desired shape using scissors or cutting blades. A tack agent is often used to keep the film in place on the fixturing. Excess film extending beyond the test specimen can easily be trimmed off before the fixturing is placed in a furnace for cure. Use of these types of adhesives results in the same amount of adhesive being used during each test, thus minimizing the influence of adhesives on transthickness strength.

**6.7.4.1** Adhesives that cure at an elevated temperature are usually sensitive to the maximum temperature; therefore, thermocouples should be attached to the fixturing (1) to insure that the cure temperature is reached and maintained, and the overall cure cycle is followed.

**NOTE 4**—Adhesives that cure at elevated temperature must reach the cure temperature in order to be activated. Extra care should be used in documenting that the temperature of the adhesive bond has been reached. It is not acceptable to simply record the temperature of the furnace and assume that the fixturing and adhesive have reached the same temperature. Improper curing of the adhesive (1) has been found to be the number one cause of bond line failures.

6.7.5 Porous CFCCs may allow the adhesive to penetrate into the interior of the CMC. Care must be taken to determine if the viscosity of the adhesive will allow it to penetrate into the test specimen. For porous CFCC systems, extra material or a spare test specimen should be bonded to blocks that are of the same material as the fixture, and then sectioned metallographically to determine the depth of penetration of the adhesive into the test specimen. The adhesive should not penetrate more than one fiber ply or more than 10 % of the specimen thickness (6) from each face.

6.8 Measurement of displacement on thicker samples can be made using a very small gage length [ $L_{GL}$ ] extensometer, strain gages, video extensometers, or noncontacting laser extensometry. No data exists to determine what effect the contacting measurement devices have on measured transthickness tensile strength. Displacement measurements can be used to calculate a transthickness elastic modulus ( $E_{ZZ}$ ) value. All displacement measurements are to be made directly on the test specimen.

**7. Precautionary Statement**

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

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

**8. Test Specimen**

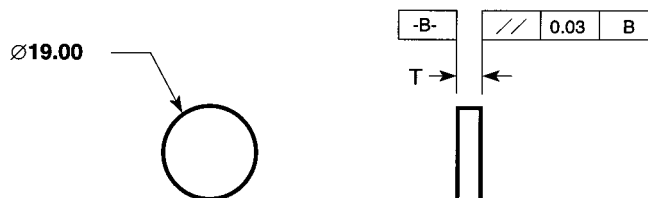
8.1 *Test Specimen Geometry:*

8.1.1 *General*—The geometry of transthickness tensile test specimens is dependent on the dimensions of the available material. For example, if the strength of an as-fabricated component is required, then the dimension of the resulting test specimen may reflect the thickness and width of the component, up to limits of the testing machine and test fixturing available. If it is desired to evaluate previously conditioned test specimens, then the size of the transthickness test specimen will be limited by the size of the conditioned test specimen. One example of a previously conditioned test specimen would be a tensile fatigue test specimen that was fatigued for a set number of cycles and the test stopped before failure occurred. A transthickness tensile test specimen could be machined out of the fatigued test specimen but would be limited in size to the width of the fatigue test specimen. Size should not be determined without the consideration of the size of the fiber and the fiber preform architecture.

8.1.1.1 The following sections discuss the most common test specimen geometries. Test specimens must have a minimum cross-sectional dimension greater than the unit cell of the fiber architecture, or a minimum of 10 mm. Any larger size is acceptable if the required forces meet the machine limitations. Deviations from the recommended geometries may be necessary depending upon the particular geometry of the available material.

8.1.1.2 Generally, circular cross-section test specimens are preferred. Test specimens using a diameter [ $D$ ] of 19 mm have been shown to provide consistent results when compared to other test specimen geometries having a similar cross-sectional area. Such test specimens generally incorporate more than two unit cells of typical fiber weaves. A typical fiber weave for CMCs is the eight harness satin weave (8HSW). An engineering drawing of a circular cross-section transthickness tensile test specimen, 19 mm in diameter, is shown in Fig. 6.

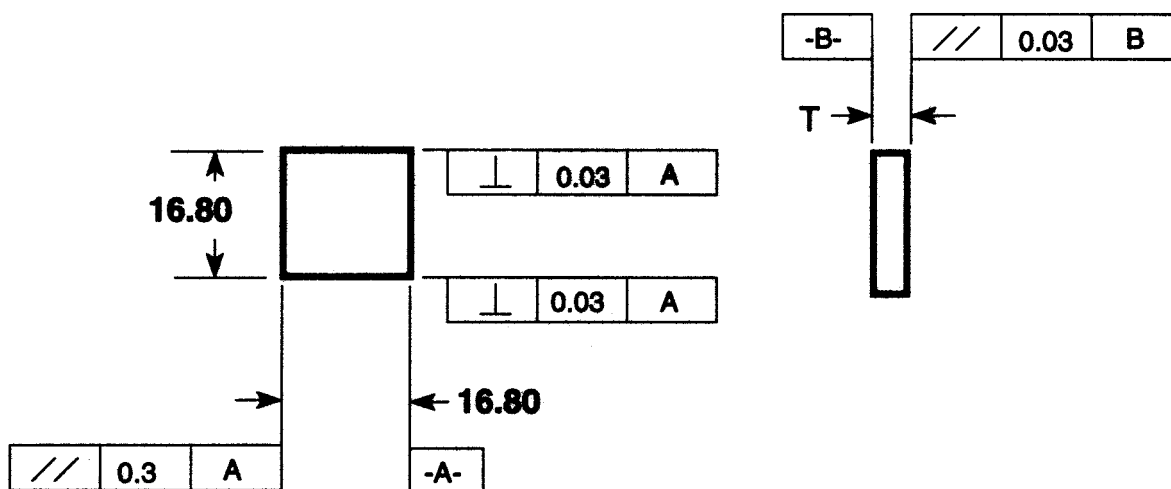
8.1.1.3 There may be instances when square or rectangular cross-section test specimens may be desirable, especially when testing sections cut out of other larger test specimens that have been conditioned or tested using other test methods. For square cross-section test specimens, a width [ $W$ ] and length [ $L$ ] of at least 16.8 mm has been shown to provide consistent results when compared to other test specimen geometries having a similar cross-sectional area (1). As the test specimen cross-sectional area is decreased, defects at the corners or edges may have more of an influence on the measured strength. For fully dense CFCC test specimens at least 16.8 mm square, the strength appears to be controlled by the microstructure of the CFCC and not the geometry of the test specimen (1). An engineering drawing of a square cross-section transthickness tensile test specimen 16.8 mm on a side is shown in Fig. 7. A dimension of 16.8 mm was selected as it is approximately the width of two unit cells of a 8HSW cloth woven produced with either silicon carbide or oxide fiber tows containing fibers with a diameter of 15  $\mu$ m or less. Both the circular and square cross-section test specimens have been used and have been shown to be effective in eliminating test specimen geometry effects for a fully dense CFCC if the cross-sectional area is maintained at approximately 282 mm<sup>2</sup> (1).



NOTE 1—Faces of test specimen can be as-processed or machined flat. All dimensions are in mm, and tolerances are: x.x $\pm$ 0.1, x.xx $\pm$ 0.03.

**FIG. 6 Drawing of a Circular Cross-Section Transthickness Tensile Test Specimen 19.0 mm in Diameter**





NOTE 1—Faces of test specimen can be as-processed or machined flat. All dimensions are in mm, and tolerances are  $x.x \pm 0.1$ ,  $x.xx \pm 0.03$ .

FIG. 7 Drawing of a Square Cross-Section Transthickness Tensile Test Specimen 16.8 mm Wide

## 8.2 Test Specimen Preparation:

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

8.2.2 *As-Fabricated*—The transthickness tensile test specimen should simulate the surface/edge conditions and processing route of an application where no machining is used; for example, as-cast, sintered, hot-pressed, or injection-molded part. No additional machining specifications are relevant.

NOTE 5—As-processed test specimens might possess rough surface textures and non-parallel edges and may be prone to premature failure if there are stress concentrations at the edges of the test specimen.

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

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

8.2.4.1 It is customary to machine only the edges of the transthickness tensile test specimen. However, the faces can be machined to make them parallel, to reduce the surface roughness, or to remove high spots. Such machining will facilitate the process of bonding the test specimen to the fixturing. It is important to note that higher surface roughness may decrease bonding integrity. In addition, machining the faces will generally damage fibers the surface plies, and any machining of the faces should be reported.

8.2.5 *Recommended Procedure*—In instances where 8.2.2-8.2.4 are not appropriate, 8.2.5 applies. Studies to evaluate the machinability of CFCCs have not been completed.

NOTE 6—Several commercial machining companies were contacted to determine the optimum procedure for machining test specimens out of CFCC material. This information has been condensed into 8.2.5. The recommended procedure of 8.2.5 can be viewed as starting-point guidelines. A more stringent procedure may be necessary.

8.2.5.1 Conduct grinding or cutting with an ample supply of appropriate filtered coolant to keep the test material and grinding wheel constantly flooded and particles flushed. Grinding can be done in at least two stages, ranging from coarse to fine rate of material removal. All cutting can be done in one stage appropriate for the depth of cut. Care must be taken during cutting and grinding to avoid “fraying” the edges of the test specimen. Fraying can be avoided by supporting one or both faces of the material and by using appropriate cutting and grinding rates. These rates will have to be determined for each CFCC system.

8.2.5.2 The cutting and grinding should be performed in an initial and final grinding operation using appropriate diamond tooling. The initial rough grinding should use a material removal rate of 0.03 mm per pass and a 180–240 grit diamond grinding wheel for the entire initial rough grinding process. Initial rough grinding should stop when 0.25 mm of material remains to be removed. Final grinding should then be performed using a material removal rate of 0.015 mm per pass and a 320–400 grit diamond wheel. If a finer finish is requested, the 400 grit diamond wheel can be substituted with a 600 grit diamond wheel.

8.2.5.3 Transthickness tension test specimens using the circular cross-section can be core drilled to an oversized diameter and diamond ground to the final dimensions using the final grinding procedure listed in 8.2.5.2.

8.2.5.4 Final grinding should be performed with the grinding wheel rotating in a plane parallel to the plies in the  $x$ - and  $y$ -directions to avoid fraying the reinforcing ceramic fibers. Machining should not be performed in the  $z$ -direction. Appropriate care should be taken to not damage the test specimen during clamping of the material.

8.2.5.5 The machined edges shall not be beveled.

8.3 *Coated Material*—CFCCs sometimes have a protective seal coat applied to the outer surface of the composite. In these instances, the coating should be removed prior to testing if determination of the transthickness tensile strength of the substrate CFCC is required. The procedures discussed in 8.2.2-8.2.5 may be used to remove this exterior coating.

8.3.1 Sometimes the seal coatings are an integral part of the CFCC, and the determination of the tensile adhesive strength between the seal coating and the substrate CFCC may be of interest. In this case, the seal coating should be retained.

8.3.2 Sufficient details regarding the coating must be included in the report. The report should list if a seal coat was originally present, whether or not it was removed, and the procedure used to remove it if applicable.

8.4 *Handling Precaution*—Exercise care in storing and handling finished test specimens to avoid the introduction of random and severe flaws. In addition, give attention to pretest storage of test specimens in controlled environments or desiccators to avoid unquantifiable environmental degradation of test specimens prior to testing.

8.5 *Number of Valid Tests*—A minimum of ten valid tests is required for the purpose of estimating a mean. A greater number of valid tests may be necessary if estimates regarding the form of the strength distribution are required. The number of valid tests required by this test method has been established with the intent of determining not only reasonable confidence limits on strength distribution parameters, but also to discern multiple fracture mechanisms. If material cost or test specimen availability limit the number of tests to be conducted, a minimum of three valid tests can be conducted to determine an indication of material properties.

8.6 *Valid Tests*—A valid individual test is one which meets all the following requirements: all the test requirements of this test method, and failure occurs within the test specimen (not at the test specimen adhesive interface, or at any point or fraction of the adhesive interface).

8.7 *Test Specimen Dimensions*—Conduct 100 % inspection/measurements of all test specimens and test specimen dimensions to assure compliance with the drawing specifications. Generally, high-resolution optical methods or high-resolution digital point contact methods are satisfactory as long as the equipment meets the specifications in 6.6.

8.7.1 Determine the thickness and width or diameter of each test specimen to within 0.02 mm. Measurements should be made on at least three different cross-sectional planes at equally spaced locations around the test specimen. To avoid damage in the critical gage-section area, make these measurements either optically or mechanically using a flat, anvil-type micrometer. In either case, the resolution of the instrument shall be as specified in 6.6. Exercise extreme caution to prevent damage to the test specimen edges. Ball-tipped or sharp-anvil micrometers are not recommended because edge damage can be induced. Record and 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.

8.7.2 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 parallel to the tensile axis. Surface roughness can have an effect on how the adhesive bonds to the test specimen. Measurement of the surface finish on the edges can provide an indication of internal defects, such as macro-porosity, which can have a very large effect on measured strength. When quantified, report surface roughness measurements.

#### 8.8 *Bonding of Test Specimens to Fixturing:*

8.8.1 It is extremely hard to grip a test specimen directly to conduct a transthickness tensile test. Therefore, fixturing must be bonded to the test specimens. This fixturing is then gripped or connected to the force train by pins and couplers. If fixed gripping is utilized, then the test specimen normally is bonded to the fixturing directly in the force frame as discussed in 6.3.2.2. Engineering drawing for fixturing that accepts a 19.0-mm circular, 16.8-mm square, and 10-mm square cross-section test specimen are shown in Figs. 8-10, respectively. The drawing are for a pin and clevis arrangement, but can be easily modified to accept a universal rod end. It is recommended that the fixturing be made out of stainless steel to minimize oxidation during adhesive cure or adhesive removal.

8.8.1.1 Thoroughly clean the mounting surfaces of the fixture. Adhesive remaining on the fixturing can easily be removed by an intermediate temperature heat treatment to char the adhesive or a diamond honing stick. After the adhesive is removed, thoroughly clean the fixturing. In some cases a very light sand blasting may be used to clean the mounting surface. Exercise care in using sandblasting, as it will slowly erode the fixturing. The fixturing will need to be refaced if they get out of tolerance. Once all adhesive and residue is removed, thoroughly clean the bonding faces of the fixturing using appropriate solvents. Appropriately dry the fixtures and store them in a desiccator until ready for assembly.

8.8.1.2 Thoroughly clean the test specimen using appropriate chemicals. Appropriately dry the test specimens and store them in a desiccator until ready for assembly. Care should be taken to not touch the faces of the test specimen with bare hands to avoid possible poisoning of the bond.

8.8.2 Alignment is extremely critical when bonding the test specimen to the fixturing. Fixed gripping systems are aligned, and only require the adhesive be applied to the fixture in the force train, and then the test specimen is placed in the force train.

8.8.3 Non-fixed force trains require that an alignment device be used when bonding the test specimen to the fixturing. A schematic of one possible alignment device and assembly hardware is shown in Figs. 11-13. A photograph showing the alignment device, spacer, test fixtures, and test specimen is shown in Fig. 14. A photograph of a second possible alignment device is shown