Designation: C 1425 – 05

Standard Test Method for Interlaminar Shear Strength of 1–D and 2–D Continuous Fiber-Reinforced Advanced Ceramics at Elevated Temperatures[1]

This standard is issued under the fixed designation C 1425; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method addresses the compression of a double-notched specimen to determine interlaminar shear strength of continuous fiber-reinforced ceramic composites (CFCCs) at elevated temperatures. Specimen preparation methods and requirements, testing modes (load or displacement control), testing rates (load rate or displacement rate), data collection, and reporting procedures are addressed.

1.2 This test method is used for testing advanced ceramic or glass matrix composites with continuous fiber reinforcement having a laminated structure such as in unidirectional (1-D) or bidirectional (2-D) fiber architecture (lay-ups of unidirectional plies or stacked fabric). This test method does not address composites with nonlaminated structures, such as (3-D) fiber architecture or discontinuous fiber-reinforced, whisker-reinforced, or particulate-reinforced ceramics.

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

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are noted in 8.1 and 8.2.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

C 1145 Terminology on Advanced Ceramics

C 1292 Test Method for Shear Strength of Continuous Fiber-Reinforced Ceramics at Ambient Temperatures

D 695 Test Method for Compressive Properties of Rigid Plastics

D 3846 Test Method for In-Plane Shear Strength of Reinforced Plastics

D 3878 Terminology for Composite Materials

E 4 Practices for Force Verification of Testing Machines

E 6 Terminology Relating to Methods of Mechanical Testing

E 122 Practice for Calculating Sample Size to Estimate, With a Specified Tolerable Error, the Average for Characteristic of a Lot or Process

E 220 Test Method for Calibration of Thermocouples by Comparison Techniques

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

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

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

3. Terminology

3.1 *Definitions*—The definitions of terms relating to shear strength testing appearing in Terminology E 6 apply to the terms used in this test method. The definitions of terms relating to advanced ceramics appearing in Terminology C 1145 apply to the terms used in this test method. The definitions of terms relating to fiber-reinforced composites appearing in Terminology D 3878 apply to the terms used in this test method.

4. Summary of Test Method

4.1 This test method addresses the determination of the interlaminar shear strength of CFCCs at elevated temperatures. The interlaminar shear strength of CFCCs, as determined by this test method, is measured by loading in compression a double-notched specimen of uniform width. Failure of the specimen occurs by interlaminar shear between two centrally located notches machined halfway through the thickness of the specimen and spaced a fixed distance apart on opposing faces. Schematics of the loading mode and the specimen are shown in

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

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

Fig. 1. The procedures in this test method are similar to those in Test Method C 1292 for the determination of the interlaminar shear strength of CFCCs at ambient temperature, except that the considerations for conducting the test at elevated temperatures are addressed in this test method.

5. Significance and Use

5.1 Continuous fiber-reinforced ceramic composites are candidate materials for structural applications requiring high degrees of wear and corrosion resistance, and damage tolerance at high temperatures.

5.2 The 1-D and 2-D CFCCs are highly anisotropic and their transthickness tensile and interlaminar shear strength are lower than their in-plane tensile and in-plane shear strength, respectively.

5.3 Shear tests provide information on the strength and deformation of materials under shear stresses.

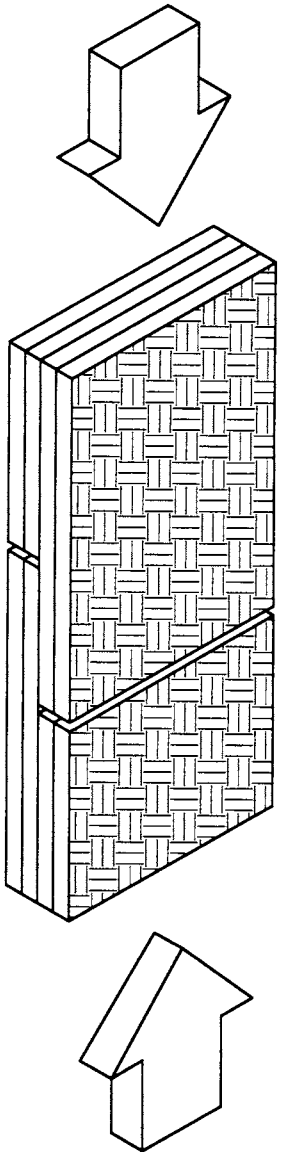


FIG. 1 Schematic of Compression of Double-Notched Specimen for the Determination of Interlaminar Shear Strength of CFCCs

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

5.5 For quality control purposes, results derived from standardized shear 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.

6. Interferences

6.1 Test environment (vacuum, inert gas, ambient air, and so forth) including moisture content (for example, relative humidity) may have an influence on the measured interlaminar shear strength. In particular, the behavior of materials susceptible to slow crack growth will be strongly influenced by test environment and testing rate. Testing to evaluate the maximum strength potential of a material shall 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 those conditions. When testing is conducted in uncontrolled ambient air with the objective 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.

6.2 Preparation of test specimens, although normally not considered a major concern with CFCCs, can introduce fabrication flaws which may have pronounced effects on the mechanical properties and behavior (for example, shape and level of the resulting load-displacement curve and shear strength). Machining damage introduced during specimen preparation can be either a random interfering factor in the determination of shear strength of pristine material, or an inherent part of the strength characteristics to be measured. Universal or standardized test methods of surface preparation do not exist. Final machining steps may, or may not, negate machining damage introduced during the initial machining. Thus, specimen fabrication history may play an important role in the measured strength distributions and shall be reported.

6.3 Bending in uniaxially loaded shear tests can cause or promote non-uniform stress distributions that may alter the desired state of stress during the test. For example, non-uniform loading will occur if the loading surfaces of the test specimen are not flat and parallel.

6.4 Fractures that initiate outside the gage section of a specimen may be due to factors such as localized stress concentrations, extraneous stresses introduced by improper loading configurations, or strength-limiting features in the microstructure of the specimen. Such non-gage section fractures will normally constitute invalid tests.

6.5 For the evaluation of the interlaminar shear strength by the compression of a double-notched specimen, the distance between the notches has an effect on the maximum load and

therefore on the interlaminar shear strength.[3,4,5] It has been found that the stress distribution in the gage section of the specimen is independent of the distance between the notches when the notches are far apart. However, when the distance between the notches is such that the stress fields around the notches interact, the measured interlaminar shear strength increases. Because of the complexity of the stress field around each notch and its dependence on the properties and homogeneity of the material, conduct a series of tests on specimens with different spacing between the notches to determine the effect of notch separation on the measured interlaminar shear strength.

6.6 For the evaluation of the interlaminar shear strength by the compression of a double-notched specimen, excessive clamping forces will reduce the stress concentration around the notches and, therefore, artificially increase the measured interlaminar shear strength. Excessive clamping might occur if interference between the fixture and the specimen results from mismatch in their thermal expansion. Section 7.6 provides guidance to prevent this problem.

6.7 The interlaminar shear strength of 1-D and 2-D CFCCs is controlled either by the matrix-rich interlaminar regions or by the weakest of the fiber-matrix interfaces. Whether interlaminar-shear failure initiates at the matrix-rich interlaminar region or at the weakest of the fiber/matrix interfaces depends on the location of the root of the notch, where the interlaminar shear stress is largest, with respect to the interlaminar microstructural features.

7. Apparatus

7.1 *Testing Machines*—The testing machine shall be in conformance with Practices E 4. The loads used in determining shear strength shall be accurate within ±1 % at any load within the selected load range of the testing machine as defined in Practices E 4.

7.2 *Heating Apparatus*—The apparatus for, and method of, heating the specimens shall provide the temperature control necessary to satisfy the requirement of 10.2.

7.2.1 Heating can be by indirect electrical resistance (heating elements), indirect induction through a susceptor, or radiant lamp with the specimen in ambient air at atmospheric pressure unless other environments are specifically applied and reported. Note that direct resistance heating is not recommended for heating CFCCs due to possible differences of the electrical resistance of the constituent materials which may produce nonuniform heating of the specimen.

7.3 *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 10.2.

7.3.1 Primary temperature measurement shall be made with thermocouples in conjunction with potentiometers, millivoltmeters, or electronic temperature controllers or readout units, or combination thereof. Such measurements are subject to two types of error. Thermocouple calibration and instrument measuring errors initially produce uncertainty as to the exact temperature. Secondly, both thermocouples and measuring instruments may be subject to variations over time. Common errors encountered in the use of thermocouples to measure temperatures include: calibration error, drift in calibration due to contamination or deterioration with use, lead-wire error, error arising from method of attachment to the specimen, direct radiation of heat to the bead, heat conduction along thermocouple wires, and so forth.

7.3.2 Temperature measurements shall be made with thermocouples of known calibration. Representative thermocouples shall be calibrated from each lot of wires used for making noble-metal (for example, platinum or rhodium) thermocouples. Except for relatively low temperatures of exposure, noble-metal thermocouples are eventually subject to error upon reuse. Oxidized noble-metal thermocouples shall not be reused without clipping back to remove wire exposed to the hot zone, re-welding, and annealing. Any reuse of noble-metal thermocouples after relatively low-temperature use without this precaution shall be accompanied by re-calibration data demonstrating that calibration was not unduly affected by the conditions of exposure.

7.3.3 Measurement of the drift in calibration of thermocouples during use is difficult. When drift is a problem during tests, a method shall be devised to check the readings of the thermocouples monitoring the specimen temperature during the test. For reliable calibration of thermocouples after use, the temperature gradient of the test furnace must be reproduced during the re-calibration.

7.3.4 Temperature-measuring, controlling, and recording instruments shall be calibrated against a secondary standard, such as precision potentiometer, optical pyrometer, or blackbody thyristor. Lead-wire error shall be checked with the lead wires in place as they normally are used. For thermocouple calibration procedures refer to Test Method E 220 and Specification E 230.

7.4 *Data Acquisition*—At a minimum, autographic records of applied load and cross-head displacement versus time shall be obtained. Either analog chart recorders or digital data acquisition systems may be used for this purpose although a digital record is recommended for ease of later data analysis. Ideally, an analog chart recorder or plotter shall be used in conjunction with the digital data acquisition system to provide an immediate record of the test as a supplement to the digital record. Recording devices must be accurate to ±1 % of full scale and shall have a minimum data acquisition rate of 10 Hz with a response of 50 Hz deemed more than sufficient.

7.5 *Dimension-Measuring Devices*—Micrometers and other devices used for measuring linear dimensions must be accurate and precise to at least 0.01 mm.

[3] Whitney, J. M., "Stress Analysis of the Double Notch Shear Specimen," *Proceedings of the American Society for Composites*, 4th Technical Conference, Blacksburg, VA, Technomic Publishing Co., Oct. 3-5, 1989, pp. 325.

[4] Fang, N. J. J., and Chou, T. W., "Characterization of Interlaminar Shear Strength of Ceramic Matrix Composites," *Journal Am. Ceram. Soc.*, 76, [10] 1993, pp. 2539-48.

[5] Lara-Curzio, E., and Ferber, M. K., "Shear Strength of Continuous Fiber Reinforced Ceramic Composites," in *Thermal and Mechanical Test Methods and Behavior of Continuous Fiber Ceramic Composites, ASTM STP 1309M*, G. Jenkins, S. T. Gonczy, E. Lara-Curzio, N. E. Ashgaugh, and L. P. Zawada, eds., American Society for Testing and Materials, Philadelphia, PA, 1996.

7.6 *Test Fixture*—The main purposes of the fixture are to allow for uniform axial compression of the specimen, and to provide lateral support to prevent buckling. Fig. 2a and 2b show schematics of test fixtures that have been used successfully to evaluate the interlaminar shear strength of CFCCs at elevated temperatures. Fig. 2a. shows the schematic of a fixture consisting of a slotted body and one loading piston. Fig. 2b shows the schematic of a fixture consisting of one hollow cylinder (sleeve), two pistons, and two semicylindrical spacers. A supporting jig conforming to the geometry of that shown in Figure 1 of Test Method D 3846 or in Figure 4 of Test Method D 695 may also be used. The material used for the manufacture of the fixture should be stable and remain rigid at the test temperature. When using a slotted-body or two semicylindrical spacers as suggested in Fig. 2a and 2b, select their dimensions so that a gap not larger than 1 % of the specimen thickness exists between the specimen and each spacer (or between the specimen and the walls of the slotted body) at the test temperature. To facilitate this requirement, use a compliant interphase between the specimen and the spacers (or walls of the slotted body). This compliant interphase will also be useful for the purpose of accommodating thermally induced deformation. To prevent mechanical interference between the fixture and the specimen and avoid compressing the specimen at the test temperature, it is recommended to manufacture the test fixture using a material with equal or higher coefficient of thermal expansion than that of the specimen in its thickness direction. To ensure uniform axial loading, the pistons should be concentric with, and form a tight clearance fit with, the sleeve or hollow cylinder (that is, the pistons should be able to slide without friction within the sleeve). This can be achieved by meeting tight cylindricity requirements for the inner diameter of the sleeve and the outer diameter of the piston.

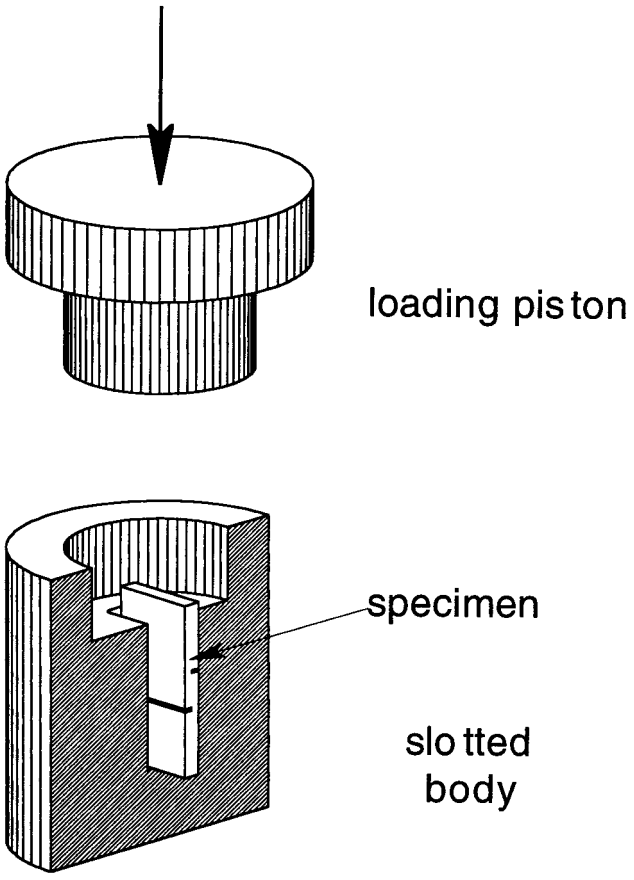


FIG. 2 Schematic of Fixture for the Compression of Double-Notched Test Specimens at Elevated Temperatures

NOTE 1—The material used to construct the fixture shall be thermochemically stable and rigid at the test temperature: (a) Sectioned view of text fixture using one piston and one slotted base (b) Cross-sectional view of fixture using two pistons and two semicylindrical spacers.

NOTE 2—0.70 mm thick aluminum-oxide paper has worked well as an interphase between 3.0-mm thick 2-D ceramic grade and Hi-Nicalon/SiC[6] CFCCs and a α-SiC fixture for tests in air at elevated temperatures. 0.79 mm thick GRAFOIL[7] has worked well as an interphase between 6.0-mm thick 1-D C/C CFCC and an aluminum-oxide fixture for tests in inert environment at elevated temperatures.[8]

8. Precautionary Statement

8.1 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.

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

9. Test Specimen

9.1 *Test Specimen Geometry*—The test specimens shall conform to the shape and tolerances shown in Fig. 3. The specimen consists of a rectangular plate with notches machined on both sides. The depth of the notches shall be at least equal to one half of the specimen thickness, and the distance between the notches shall be determined considering the requirements to produce shear failure in the gage section. Furthermore, because the measured interlaminar shear strength may be dependent on the notch separation, it is recommended to conduct tests with different values of notch separation to determine this dependence. The edges of the specimens shall be smooth, but not rounded or beveled. Table 1 contains recommended values for the dimensions associated with the specimen shown in Fig. 3.

NOTE 3—Because many CFCCs are produced as flat plates and the outer surfaces may reflect the texture of the underlying fiber bundles, as-fabricated plates might not meet the parallelism requirements prescribed in Fig. 3 without additional machining of the specimen faces. The faces of the specimens shall not deviate from parallelism by more than

[6] Hi-Nicalon/SiC, a registered trademark of UCAR Carbon Company, Inc. P. O. Box 218, Columbia, TN 38402-0218, has been found satisfactory for this purpose.

[7] GRAFOIL, a registered trademark a registered trademark of UCAR Carbon Company, Inc. P. O. Box 218, Columbia, TN 38402-0218, has been found satisfactory for this purpose.

[8] Lara-Curzio, E., Bowers, David, and Ferber, M. K., "The Interlaminar Tensile and Shear Properties of a Unidirectional C/C Composite," *Journal of Nuclear Materials*, 230, 1996, pp. 226-32.

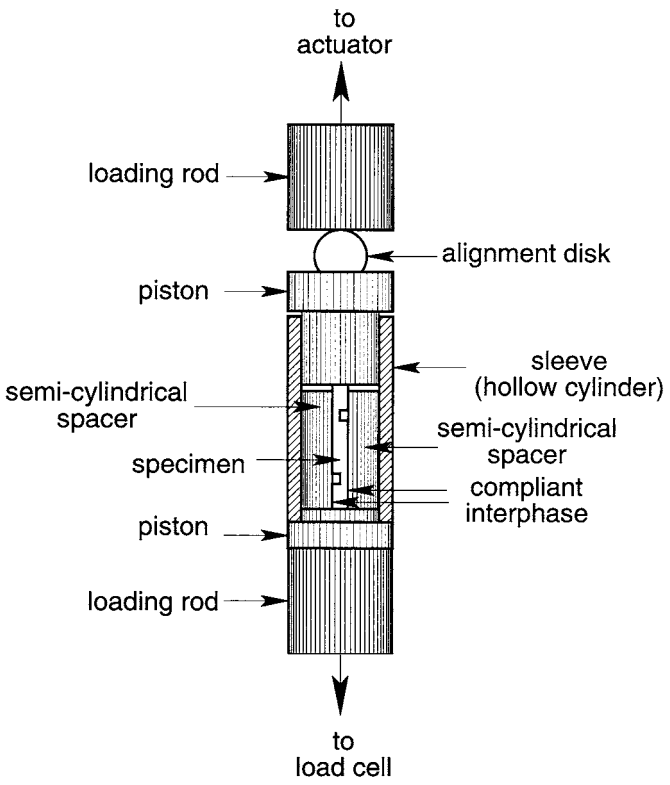
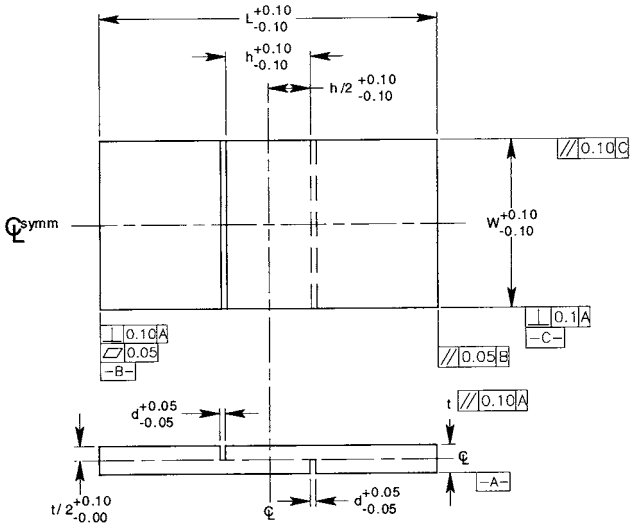


FIG. 3 Schematic of Fixture for the Compression of Double-Notched Test Specimens at Elevated Temperatures



NOTE 1—All tolerances are in millimetres. Refer to Table 1.

FIG. 4 Dimensions of Double-Notched Test Specimen

5 % of the average thickness of the specimen if it is impractical to machine the specimen faces to meet the parallelism requirements in Fig. 3.

NOTE 4—Although in practice it is impossible to obtain a perfectly square notch as suggested in Fig. 3, efforts should be made during sample preparation to minimize rounding the bottom of the notch. This can be accomplished, for example, by frequently dressing the wheel used to machine the notches since wear will tend to round its tip. At this time, studies of the effect of notch shape on the interlaminar shear strength of CFCCs have not been completed.

TABLE 1 Recommended Dimensions for Double-Notched Compression Specimen

Dimension	Description	Value, mm	Tolerance, mm
L	specimen length	30.00	±0.10
h	distance between notches	6.00	±0.10
W	specimen width	15.00	±0.10
d	notch width	0.50	±0.05
t	specimen thickness	—	—

9.2 *Specimen Preparation*:

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

9.2.2 *Standard Procedures*—Studies to evaluate the machinability of CFCCs have not been completed. Therefore, the standard procedure of this section can be viewed as starting-point guidelines but a more stringent procedure may be necessary.

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

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

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

9.4 *Number of Test Specimens*—A minimum of 10 test specimens per test condition shall be tested, unless valid results can be gained through the use of fewer specimens, such as in the case of a designed experiment. For statistically significant data, the procedures outlined in Practice E 122 shall be consulted.

10. Procedure

10.1 *Specimen Dimensions*—Determine the width of the gage section of each specimen and the distance between the notches to within 0.02 mm. Avoid damaging the critical gage section area by performing 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 must be as specified in 7.5. Exercise extreme caution to prevent damaging the specimen gage section. Record and report the measured dimensions and locations of the measurements for use in the calculation of the shear stress. For example, measure the width of the specimen at the location of the notches and at the middle of the gage section, and use the average of multiple measurements in the stress calculations. Measure the notch separation on both edges of the specimen and use the average of these measurements in the stress calculations.

NOTE 5—It has been found that an optical comparator works best to measure the distance between the notches.

10.2 *Temperature Control*—Form the thermocouple bead in accordance with the Preparation of Thermocouple Measuring Junctions[9]. Generally, noble-metal (for example, platinum or rhodium) thermocouples shall not be attached directly to CFCC materials due to chemical incompatibility. The thermocouple junction may be brought close to the specimen (3 to 6 mm) and shielded. Shielding may be omitted if, for a particular furnace, the difference in indicated temperature from an unshielded bead and a bead inserted in a hole in the specimen has been shown to be less than one half the variation listed in 10.2.2. The bead shall be as small as possible and there shall be no shorting of the circuit (such as could occur from twisted wire behind the bead). Use ceramic insulators on the thermocouples in the hot zone. If some other electrical insulation material is used in the hot zone, it shall be carefully checked to determine whether the electrical insulating properties are maintained at higher temperatures.

10.2.1 *Number of Required Thermocouples*—Employ at least two thermocouples, one near each end of the gage section.

NOTE 6—If it is possible to insert the thermocouples into the fixture and position their tip close to the specimen then do so. If the furnace is large enough so that the entire fixture and test specimen can be maintained at the same test temperature, then place the thermocouples next to the fixture at the location of the edges of the gage section.

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

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

In addition, temperature variation within the uniformly heated gage section shall not exceed the following:

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

10.2.3 The term "indicated temperature" means temperature that is indicated by the temperature measuring device using good quality pyrometric practice. It is recognized that true temperature may vary more than the indicated temperature. The permissible indicated temperature variations of 10.2.2 are not to be construed as minimizing the importance of good pyrometric practice and precise temperature control. All laboratories shall keep both indicated and true temperature variations as small as practicable. It is recognized that in view of the dependency of creep deformation of materials on temperature, close temperature measurement is necessary. The limits prescribed represent ranges which are common practice.

10.2.4 Temperature overshoots during heating shall not exceed the limits stated in 10.2.2. The heating characteristics of the furnace and the temperature control system shall be studied to determine the power input, temperature set point, proportioning control adjustment, and control-thermocouple placement to limit transient temperature overshoots. It may be desirable to stabilize the furnace at a temperature 10 to 25 K less than the nominal test temperature before making the final adjustments. Report any temperature overshoots with details of magnitude and duration.

10.2.5 *Temperature Rates and Hold Time*—The rate at which temperature can be increased from ambient to the final test temperature depends on many factors, such as, heating system, temperature controller, test material, and test environment. Limiting time at the test temperature will minimize time-dependent thermal or environmental degradation, or both. In addition, some materials experience so-called oxidation due to "low-temperature chemical instabilities" which occur at intermediate temperatures. With these materials, the temperature ramp shall be as rapid as possible to minimize the exposure time to these intermediate temperatures. Generally, good results have been obtained for heating rates in which the specimen temperature is ramped from ambient to the test temperature at a constant rate between 30 K/min and 60 K/min. The hold time at temperature prior to the start of the test shall be governed by the time necessary to ensure that the specimen has reached equilibrium and that the temperature can be maintained within the limits specified in 10.2.2. Report both the time to attain test temperature and the time at temperature before loading.

NOTE 7—Some CFCCs rely on the formation of oxide layers or on the flow of low-viscosity phases for sealing and protecting the interior of the composite by preventing the ingression of the service environment (for example, oxidizing) at elevated temperatures. However, severe environmental degradation of some CFCCs has been documented (at temperatures as low as 573 K) when the service environment (for example, oxidizing) is allowed to ingress to the interior of the composite at temperatures where the formation of a protective oxide layer or the flow of glassy coatings is inhibited. This is particularly true for CFCCs that rely on the integrity of C and BN fiber coatings, and SiC-based fibers to promote composite behavior.

10.3 *Test Modes and Rates*:

10.3.1 *General*—Test modes may involve load or displacement control. Recommended rates of testing must be sufficiently rapid to obtain the maximum possible shear strength at fracture of the material within 30 s. However, rates other than those recommended here may be used to evaluate rate effects. In all cases the test mode and rate must be reported.

10.3.1.1 Generally, displacement 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. However, for sufficiently rapid test rates, differences in the fracture process may not be noticeable and any of these test modes may be appropriate.

10.3.2 *Displacement Rate*—Use a constant cross-head displacement rate of 0.02 mm/s unless otherwise found acceptable as determined in 10.3.1 or 10.3.1.1.

10.3.3 *Load Rate*—Select a constant load rate to produce final fracture in 10 to 30 s or to be approximately equivalent to a test rate of 0.02 mm/s.

10.4 *Preparations for Testing*—Set the test mode and test rate on the test machine. Ready the autograph data acquisition systems for data logging.

10.5 *Conducting the Test*:

[9] *1982 Annual Book of ASTM Standards*, Part 44, Related Materials Section.

10.5.1 Mount the specimen in the test fixture.

10.5.2 *Preparations for Testing*—Set the test mode and test rate on the test machine. Pre-load the specimen to remove the slack from the load train. The amount of pre-load, which shall not exceed 10 % of the test load, will depend on the material and shall be reported for each situation. Ready the autograph data acquisition systems for data logging. Begin recording furnace temperature when furnace heating is initiated and continue recording until the completion of the test. Maintain a constant minimal force in the load train to allow for the thermal expansion of the specimen and load train during specimen heat up. It is recommended to use a test machine that allows for the control of the load during heating up and during mechanical loading. Heat the specimen to the test temperature at the prescribed heating rate and hold constant at temperature until the specimen reaches thermal equilibrium.

10.5.3 Initiate data collection. Load the specimen to failure at the prescribed loading rate.

10.5.4 After specimen fracture, disable the action of the test machine and the data collection of the data acquisition system. The breaking load should be measured with an accuracy of ±1 % of the load range and noted for the report. Retract the cross-head or actuator, and allow the furnace to cool down. Carefully remove the specimen from the fixture. Avoid damaging the fracture surfaces by preventing them from contacting each other or other objects.

10.5.5 Determine the relative humidity in accordance with Test Method E 337.

10.5.6 Note that the use of results from specimens fracturing outside the gage section cannot be used in the direct calculation of a mean shear strength. Results from specimens fracturing outside the gage section are considered anomalous and can be used only as censored tests. To complete a required statistical sample for purposes of average strength, one replacement specimen should be tested for each specimen which fractures outside the gage section.

10.5.7 Visual examination and optical microscopy are recommended to determine the mode and type of fracture, as well as the location of fracture initiation.

11. Calculation

11.1 *Shear Strength*—Calculate the shear strength as follows:

$$\text{Shear Strength} = \frac{P_{max}}{A} \tag{1}$$

where:

P_{max} = applied maximum load, and
A = average shear stressed area, which is calculated as:

$$A = Wh \tag{2}$$

where:

W = average specimen width, and
h = average distance between the notches (Fig. 3) as described in 10.1.

11.2 *Statistics*—For each series of tests, calculate the average value, standard deviation, and coefficient of variation (in percent) for each property determined:

$$\bar{x} = \frac{1}{n}\left(\sum_{i=1}^{n} x_i\right) \tag{3}$$

$$S_{n-1} = \sqrt{\left(\sum_{i=1}^{n} x_i^2 - n\bar{x}^2\right) / (n-1)} \tag{4}$$

$$CV = 100\,(S_{n-1} / \bar{x}) \tag{5}$$

where:

$\bar{x}$ = sample mean (average),
S_{n-1} = sample standard deviation,
CV = sample coefficient of variation, %,
n = number of specimens, and
x_i = measured or derived property.

12. Report

12.1 *Test Set*—Report the following information for the test set. Any significant deviations from the procedures and requirements of this test method shall be noted in the report.

12.1.1 Date and location of testing.

12.1.2 Test specimen geometry used (include engineering drawing).

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

12.1.4 A drawing or sketch of the type and configuration of the specimen mount.

12.1.5 The total number of specimens (n) with special emphasis on the number of specimens that fractured in the gage section. This information will reveal the success rate of the particular specimen geometry and test apparatus.

12.1.6 All relevant data such as vintage and identification data, with emphasis on the date of manufacture of the material and a short description of reinforcement (type, layup, and so forth), fiber volume fraction, and bulk density. For commercial materials, the commercial designation must be reported.

12.1.6.1 For noncommercial materials, the major constituents and proportions must be reported as well as the primary processing route including green state and consolidation routes. Also report fiber volume fraction, matrix porosity, and bulk density.

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

12.1.8 Heat treatments, coatings, or pretest exposures, if any, applied either to the as-processed material or to the as-fabricated specimen.

12.1.9 Test environment including relative humidity (Test Method E 337) and atmosphere (for example ambient air, dry nitrogen, and so forth).

12.1.10 The heating rate, test temperature, time at temperature, duration of the test, and time to cool to ambient temperature after the completion of the test.

12.1.11 Test mode (load or displacement control) and actual test rate (load rate or displacement rate).

12.1.12 Pre-load (if used) to heat up the specimen to the test temperature.

12.1.13 Specimen dimensions, that is, average notch separation and average width.

12.1.14 Mean, standard deviation, and coefficient of variation for the measured shear strength for each test series.

12.1.15 Appearance of specimen after fracture.

13. Precision and Bias

13.1 Because of the nature of these materials and the 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 this test method.

14. Keywords

14.1 composite; compression; continuous fiber-reinforced ceramic composite (CFCC); interlaminar; shear; shear strength