



# Standard Practice for Constant-Amplitude, Axial, Tension-Tension Cyclic Fatigue of Continuous Fiber-Reinforced Advanced Ceramics at Ambient Temperatures<sup>1</sup>

This standard is issued under the fixed designation C 1360; 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 practice covers the determination of constant-amplitude, axial tension-tension cyclic fatigue behavior and performance of continuous fiber-reinforced advanced ceramic composites (CFCCs) at ambient temperatures. This practice builds on experience and existing standards in tensile testing CFCCs at ambient temperatures and addresses various suggested test specimen geometries, specimen fabrication methods, testing modes (force, displacement, or strain control), testing rates and frequencies, allowable bending, and procedures for data collection and reporting. This practice does not apply to axial cyclic fatigue tests of components or parts (that is, machine elements with nonuniform or multiaxial stress states).

1.2 This practice 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 practice may also be used with glass (amorphous) matrix composites with 1-D, 2-D, 3-D, and other multi-directional continuous fiber reinforcements. This practice does not directly address discontinuous fiber-reinforced, whisker-reinforced or particulate-reinforced ceramics, although the 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 IEEE/ASTM SI 10 Standard.

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:

<sup>1</sup> This practice 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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C 1145 Terminology of Advanced Ceramics<sup>2</sup>

C 1275 Test Method for Monotonic Tensile Strength Testing of Continuous Fiber-Reinforced Advanced Ceramics with Solid Rectangular Cross-Section Specimens at Ambient Temperatures<sup>2</sup>

D 3479 Test Methods for Tension-Tension Fatigue of Oriented Fiber, Resin Matrix Composites<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 83 Practice for Verification and Classification of Extensometers<sup>4</sup>

E 337 Test Method for Measured Humidity with Psychrometer (Measurement of Wet-and Dry-Bulb Temperatures)<sup>5</sup>

E 467 Practice for Verification of Constant Amplitude Dynamic Loads in an Axial Load Fatigue Testing Machine<sup>6</sup>

E 468 Practice for Presentation of Constant Amplitude Fatigue Test Results for Metallic Materials<sup>6</sup>

E 739 Practice for Statistical Analysis of Linear or Linearized Stress-Life (S-N) and Strain-Life ( $\epsilon$ -N) Fatigue Data<sup>6</sup>

E 1012 Practice for Verification of Specimen Alignment Under Tensile Loading<sup>4</sup>

E 1823 Terminology Relating to Fatigue and Fracture Testing<sup>4</sup>

IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI) (The Modern Metric System)<sup>7</sup>

## 3. Terminology Definitions

### 3.1 Definitions:

3.1.1 Definitions of terms relating to advanced ceramics, fiber-reinforced composites, tensile testing, and cyclic fatigue as they appear in Terminology C 1145, Terminology D 3878, Terminology E 6, and Terminology E 1823, respectively, apply

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

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

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

<sup>5</sup> Annual Book of ASTM Standards, Vol 07.02.

<sup>6</sup> Annual Book of ASTM Standards, Vol 14.01.

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

to the terms used in this practice. Selected terms with definitions non specific to this practice follow in 3.2 with the appropriate source given in parenthesis. Terms specific to this practice are defined in.

3.2 Definitions of Terms Non Specific to This Standard:

3.2.1 *advanced ceramic, n*—A highly engineered, high performance predominately non-metallic, inorganic, ceramic material having specific functional attributes. (See Terminology C 1145.)

3.2.2 *axial strain [LL<sup>-1</sup>], n*—the average longitudinal strains measured at the surface on opposite sides of the longitudinal axis of symmetry of the test specimen by two strain-sensing devices located at the mid length of the reduced section. (See Practice E 1012.)

3.2.3 *bending strain [LL<sup>-1</sup>], 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 test specimen. (See Practice E 1012.)

3.2.4 *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 (See Test Method C 1275.)

3.2.5 *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. (See Terminology C 1145.)

3.2.6 *constant amplitude loading, n*—in cyclic fatigue loading, a loading in which all peak loads are equal and all of the valley loads are equal. (See Terminology E 1823.)

3.2.7 *cyclic fatigue, n*—the process of progressive localized permanent structural change occurring in a material subjected to conditions that produce fluctuating stresses and strains at some point or points and that may culminate in cracks or complete fracture after a sufficient number of fluctuations. (See Terminology E 1823.) See Fig. 1 for nomenclature relevant to cyclic fatigue testing.

3.2.7.1 *Discussion*—In glass technology static tests of considerable duration are called “static fatigue” tests, a type of test

generally designated as stress-rupture.

3.2.7.2 *Discussion*—Fluctuations may occur both in force and with time (frequency) as in the case of “random vibration.”

3.2.8 *cyclic fatigue life, N<sub>f</sub>*—the number of loading cycles of a specified character that a given test specimen sustains before failure of a specified nature occurs. (See Terminology E 1823.)

3.2.9 *cyclic fatigue limit, S<sub>f</sub> [FL<sup>-2</sup>], n*—the limiting value of the median cyclic fatigue strength as the cyclic fatigue life, N<sub>f</sub>, becomes very large, (for example, N<sub>f</sub> 10<sup>6</sup>–10<sup>7</sup>). (See Terminology E 1823.)

3.2.9.1 *Discussion*—Certain materials and environments preclude the attainment of a cyclic fatigue limit. Values tabulated as “fatigue limits” in the literature are frequently (but not always) values of S<sub>f</sub> at 50 % survival at N<sub>f</sub> cycles of stress in which the mean stress, S<sub>m</sub>, equals zero.

3.2.10 *cyclic fatigue strength S<sub>N</sub>, [FL<sup>2</sup>], n*—the limiting value of the median cyclic fatigue strength at a particular cyclic fatigue life, N<sub>f</sub>. (See Terminology E 1823.)

3.2.11 *gage length, [L], n*—the original length of that portion of the test specimen over which strain or change of length is determined. (See Terminology E 6.)

3.2.12 *force ratio, n*—in cyclic fatigue loading, the algebraic ratio of the two loading parameters of a cycle; the most widely used ratios (See Terminology E 1150):

$$R = \frac{\text{minimum force}}{\text{maximum force}} \text{ or } R = \frac{\text{valley force}}{\text{peak force}}$$

and

$$A = \frac{\text{force amplitude}}{\text{mean force}} \text{ or } A = \frac{(\text{maximum force} - \text{minimum force})}{(\text{maximum force} + \text{minimum force})}$$

3.2.13 *matrix-cracking stress [FL<sup>-2</sup>], n*—The applied tensile stress at which the matrix cracks into a series of roughly parallel blocks normal to the tensile stress. (See Test Method C 1275.)

3.2.13.1 *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 that 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 curve (elastic limit).

3.2.14 *modulus of elasticity [FL<sup>-2</sup>], n*—The ratio of stress to corresponding strain below the proportional limit. (See Terminology E 6.)

3.2.15 *proportional limit stress [FL<sup>-2</sup>], n*—the greatest stress that a material is capable of sustaining without any deviation from proportionality of stress to strain (Hooke’s law). (See Terminology E 6.)

3.2.15.1 *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, specify the procedure and sensitivity of the test equipment.

3.2.16 *percent bending, n*—the bending strain times 100 divided by the axial strain. (See Practice E 1012.)

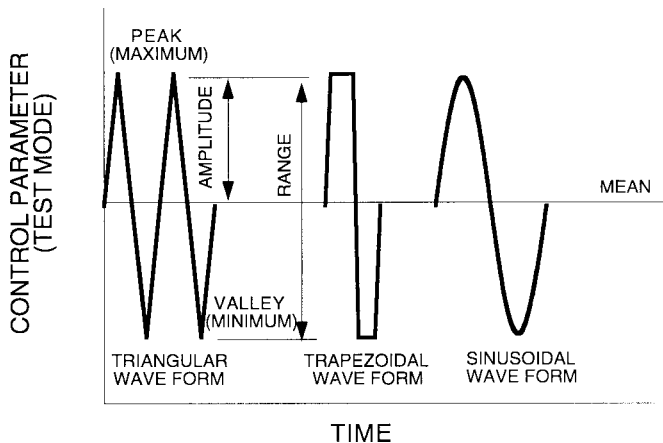


FIG. 1 Cyclic Fatigue Nomenclature and Wave Forms.

3.2.17 *S-N diagram, n*—a plot of stress versus the number of cycles to failure. The stress can be maximum stress,  $S_{max}$ , minimum stress,  $S_{min}$ , stress range,  $\Delta S$  or  $S_r$ , or stress amplitude,  $S_a$ . The diagram indicates the *S-N* relationship for a specified value of  $S_m$ ,  $A$ ,  $R$  and a specified probability of survival. For  $N$ , a log scale is almost always used, although a linear scale may also be used. For  $S$ , a linear scale is usually used, although a log scale may also be used. (See Terminology E 1150 and Practice E 468.)

3.2.18 *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 (See Test Method C 1275).

3.2.19 *tensile strength [FL<sup>-2</sup>], 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. (See Terminology E 6.)

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

3.3.1 *fracture strength [FL<sup>-2</sup>], n*—the tensile stress that the material sustains at the instant of fracture. Fracture strength is calculated from the force at fracture during a tension test carried to rupture and the original cross-sectional area of the test specimen.

3.3.1.1 *Discussion*—In some cases, the fracture strength may be identical to the tensile strength if the force at fracture is the maximum for the test.

3.3.2 *maximum stress, S<sub>max</sub>[FL<sup>-2</sup>], n*—the maximum applied stress during cyclic fatigue.

3.3.3 *mean stress, S<sub>a</sub>[FL<sup>-2</sup>], n*—the difference between the mean stress and the maximum or minimum stress such that

$$S_m = \frac{S_{max} + S_{min}}{2} \quad (1)$$

3.3.4 *minimum stress, S<sub>min</sub>[FL<sup>-2</sup>], n*—the minimum applied stress during cyclic fatigue.

3.3.5 *stress amplitude, S<sub>a</sub>[FL<sup>-2</sup>], n*—the difference between the mean stress and the maximum stress such that

$$S_a = \frac{S_{max} - S_{min}}{2} = S_{max} - S_m = S_m - S_{min} \quad (2)$$

3.3.6 *stress range, ΔS or S<sub>r</sub>[FL<sup>-2</sup>], n*—the difference between the maximum stress and the minimum stress such that

$$\Delta S = S_r = S_{max} - S_{min} \quad (3)$$

3.3.7 *time to cyclic fatigue failure, t<sub>f</sub> [t], n*—total elapsed time from test initiation to test termination required to reach the number of cycles to failure.

## 4. Significance and Use

4.1 This practice 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 are generally characterized by crystalline matrices and ceramic fiber reinforcements. These materials are candidate materials for structural applications requiring high degrees of wear and corrosion resistance, and high-temperature inherent damage tolerance (that is, toughness). In addition, continuous fiber-reinforced glass matrix composites are candidate materials for

similar but possibly less-demanding applications. Although flexural test methods are commonly used to evaluate the mechanical behavior of monolithic advanced ceramics, the non-uniform stress distribution in a flexural test specimen in addition to dissimilar mechanical behavior in tension and compression for CFCCs leads to ambiguity of interpretation of test results obtained in flexure for CFCCs. Uniaxially-loaded tensile tests provide information on mechanical behavior for a uniformly stressed material.

4.3 The cyclic fatigue behavior of CFCCs can have appreciable non-linear effects (for example, sliding of fibers within the matrix) which may be related to the heat transfer of the specimen to the surroundings. Changes in test temperature, frequency, and heat removal can affect test results. It may be desirable to measure the effects of these variables to more closely simulate end-use conditions for some specific application.

4.4 Cyclic fatigue by its nature is a probabilistic phenomenon as discussed in STP 91A<sup>8</sup> and STP 588<sup>9</sup>. In addition, the strengths of the brittle matrices and fibers of CFCCs are probabilistic in nature. Therefore, a sufficient number of test specimens at each testing condition is required for statistical analysis and design, with guidelines for sufficient numbers provided in STP 91A<sup>8</sup>, STP 588<sup>9</sup>, and Practice E 739. Studies to determine the influence of test specimen volume or surface area on cyclic fatigue strength distributions for CFCCs have not been completed. The many different tensile test specimen geometries available for cyclic fatigue testing may result in variations in the measured cyclic fatigue behavior of a particular material due to differences in the volume of material in the gage section of the test specimens.

4.5 Tensile cyclic fatigue tests provide information on the material response under fluctuating uniaxial tensile stresses. Uniform stress states are required to effectively evaluate any nonlinear stress-strain behavior which may develop as the result of cumulative damage processes (for example, matrix microcracking, fiber/matrix debonding, delamination, cyclic fatigue crack growth, etc.)

4.6 Cumulative damage due to cyclic fatigue may be influenced by testing mode, testing rate (related to frequency), differences between maximum and minimum force ( $R$  or  $A$ ), effects of processing or combinations of constituent materials, and/or environmental influences (including test environment and pre-test conditioning), or both. Some of these effects may be consequences of stress corrosion or sub critical (slow) crack growth which can be difficult to quantify. Other factors which may influence cyclic fatigue behavior are: matrix or fiber material, void or porosity content, methods of test specimen preparation or fabrication, volume percent of the reinforcement, orientation and stacking of the reinforcement, test specimen conditioning, test environment, force or strain limits during cycling, wave shapes (that is, sinusoidal, trapezoidal, etc.), and failure mode of the CFCC.

<sup>8</sup> A Guide for Fatigue Testing and The Statistical Analysis of Fatigue Data, ASTM STP 91 A, ASTM, 1963. Alternative reference: Fatigue Data Analysis, R.C. Rice, in ASM Handbook, Vol 8, 1985, pp. 695-720.

<sup>9</sup> Manual on Statistical Planning and Analysis for Fatigue Experiments, ASTM STP 588, ASTM, 1975.

4.7 The results of cyclic fatigue 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 cyclic fatigue behavior of the entire, full-size end product or its in-service behavior in different environments.

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

4.9 The cyclic fatigue behavior of a CFCC is dependent on its inherent resistance to fracture, the presence of flaws, or damage accumulation processes, or both. There can be significant damage in the CFCC test specimen without any visual evidence such as the occurrence of a macroscopic crack. This can result in a loss of stiffness and retained strength. Depending on the purpose for which the test is being conducted, rather than final fracture, a specific loss in stiffness or retained strength may constitute failure. In cases where fracture occurs, analysis of fracture surfaces and fractography, though beyond the scope of this practice, 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 cyclic fatigue behavior. In particular, the behavior of materials susceptible to slow crack growth fracture will be strongly influenced by test environment and testing rate. 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. Regardless of whether testing is conducted in uncontrolled ambient air or controlled environments, monitor and report relative humidity and temperature at a minimum at the beginning and end of each test, and hourly (if possible) if the test duration is greater than 1 h. Testing at humidity levels greater than 65 % relative humidity (RH) is not recommended.

5.2 Rate effects in many CFCCs may play important roles in degrading cyclic fatigue performance. In particular, high testing rates (that is, high frequency) may cause localized heating due to frictional sliding of debonded fibers within the matrix. Such sliding may accelerate mechanical degradation of the composite leading to rapid cyclic fatigue failures. Conversely, low testing rates (that is, low frequency or wave forms with plateaus) may serve to promote environmental degradation as the material is exposed to maximum tensile stresses for longer periods of time.

5.3 In many materials, amplitude of the cyclic wave form is a primary contributor to the cyclic fatigue behavior. Thus, choice of force ratio,  $R$  or  $A$ , can have a pronounced effect on the cyclic fatigue behavior of the material. A force ratio of  $R = 1$  (that is, maximum equal to minimum) constitutes a constant force test with no fluctuation of force over time. A force ratio of  $R = 0$  (that is, minimum equal to zero) constitutes the maximum amplitude (that is, amplitude equal to one half the maximum) for tension-tension cyclic fatigue. A force ratio of  $R$

$= 0.1$  is often chosen for tension-tension cyclic fatigue so as to impose maximum amplitudes while minimizing the possibility of a “slack” (that is, loose and non-tensioned) force train. The choice of  $R$  or  $A$  is dictated by the final use of the test result.

5.4 Surface preparation of test specimens, although normally not considered a major concern in CFCCs, can introduce fabrication flaws which may have pronounced effects on cyclic fatigue behavior (for example, shape and level of the resulting stress-strain curves, cyclic fatigue limits, etc.). Machining damage introduced during test specimen preparation can be either a random interfering factor in the determination of cyclic fatigue or ultimate strength of pristine material (that is, more frequent occurrence 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 test specimen faces without compromising the in-plane fiber architecture). Note that final machining steps may, or may not, negate machining damage introduced during the initial machining. Thus, report test specimen fabrication history since it may play an important role in the cyclic fatigue behavior.

5.5 Bending in uniaxial tensile tests can cause or promote non-uniform stress distributions with maximum stresses occurring at the test specimen surface leading to non-representative fractures originating at surfaces or near geometrical transitions. In addition, if deformations or strains are measured at surfaces where maximum or minimum stresses occur, bending may introduce over or under measurement of strains depending on the location of the strain-measuring device on the test specimen. Similarly, fracture from surface flaws may be accentuated or suppressed by the presence of the non-uniform stresses caused by bending.

5.6 Fractures that initiate outside the uniformly-stressed gage section of a test 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 test specimen. Such non-gage section fractures will normally constitute invalid tests. In addition, for face-forced 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 may initiate fracture in the vicinity of the grips.

## 6. Apparatus

6.1 *Tensile Testing Machines*—Machines used for determining proportional limit stress, ultimate strength or other “static” material properties shall conform to Practices E 4. Machines used for cyclic fatigue testing may be either nonresonant mechanical, hydraulic, or magnetic systems or resonant type using forced vibration excited by magnetic or centrifugal force and shall conform to Practice E 467.

6.2 *Gripping Devices*—Devices used to grip the test specimens may be of the types discussed in 6.2 of Test Method



C 1275 as long as they meet the requirements of this practice and Test Method C 1275.

**6.3 Load Train Couplers**—Devices used to align the load train and to act as an interface between the gripping devices and the testing machine may be of the types discussed in 6.3 of Test Method C 1275 as long as they meet the requirements of this practice and Test Method C 1275.

**6.4 Strain Measurement**—Determine strain by means of either a suitable extensometer or strain gages as discussed in Test Method C 1275. Extensometers shall satisfy Practice E 83, Class B-1 requirements and are recommended instead of strain gages for test specimens with gage lengths of  $\geq 25$  mm. Calibrate extensometers periodically in accordance with Practice E 83.

**6.5 Allowable Bending**—Analytical and empirical studies of the effect of bending on the cyclic fatigue behaviour of CFCCs do not exist. Until such information is forthcoming for CFCCs, this practice adopts the recommendations of Test Method C 1275. However, note that unless all test specimens are properly strain gaged and percent bending is monitored during testing, there will be no record of percent bending for each test specimen. Therefore, verify the testing system using the procedures detailed in Practice E 1012 and Test Method C 1275 such that percent bending does not exceed five at a mean strain equal to either one half of 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. Conduct the verification at a minimum at the beginning and end of each test series as recommended in Test Method C 1275. An additional verification of alignment is recommended, although not required, at the middle of the test series. In addition, plot a curve of percent bending vs. the test parameter (force, displacement, strain, etc.) to assist in determining the role of bending over the course of the wave form from the minimum to the maximum.

**6.6 Data Acquisition**—If desired, obtain an autographic record of applied force and gage section elongation or strain versus time at discrete periods during cyclic fatigue testing. 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 1.0 % of the recording range and shall have minimum data sampling and acquisition rates sufficient to adequately describe the loading cycle (for example,  $\sim 100$  data points per cycle).

**6.7 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. Measure cross-sectional dimensions to within 0.02 mm using dimension-measuring devices with accuracies of 0.01 mm.

**6.8 Temperature Measurement**—Cyclic fatigue tests may be run at high cyclic frequencies ( $>50$  Hz) that can cause internal heating of the test specimen thereby affecting the cyclic fatigue

life especially in the case of debonded and sliding fibers<sup>10</sup>. If test specimen heating is likely to occur or when there is doubt, monitor the test specimen temperature during the cycling. Possible methods are: the use of radiation thermometer, thermocouples adhered to the specimen, or optical pyrometry.

**6.8.1 Environmental Conditions**—For ambient temperature tests conducted under constant environmental conditions, control temperature and relative humidity to within  $\pm 3^\circ\text{C}$  and  $\pm 10$  % RH, respectively. Measure and report temperature and relative humidity in accordance with 9.3.5.

## 7. Precautionary Statement

**7.1** During the conducting of this practice, 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 safety purposes as well as later fractographic reconstruction and analysis are 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**—Tensile test specimens as discussed in 8.1 of Test Method C 1275 may be used for cyclic fatigue testing as long as they meet the requirements of this practice and Test Method C 1275.

**8.2 Test Specimen Preparation**—Test specimen fabrication and preparation methods as discussed in 8.2 of Test Method C 1275 may be used for cyclic fatigue testing as long as they meet the requirements of this practice and Test Method C 1275.

**8.3 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 pre-test storage of test specimens in controlled environments or desiccators to avoid unquantifiable environmental degradation of test specimens prior to testing. If conditioning is required, Test Methods D 3479 recommends conditioning and testing polymeric composite test specimens in a room or enclosed space maintained at a temperature and relative humidity of  $23 \pm 3^\circ\text{C}$  and  $65 \pm 10$  %, respectively. Measure ambient conditions in accordance with Test Method E 337.

**8.4 Number of Test Specimens**—The number of test specimens will depend on the purpose of the particular test. Refer to STP 91-A as a guide to determining the number of test specimens and statistical methods.

**8.5 Valid Tests**—A valid individual test is one that meets all the following requirements: all the testing requirements of this Practice and Test Method C 1275, and for a test involving a failed test specimen, 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.

<sup>10</sup> J.W. Holmes X. Wu, B.F. Sorensen, "Frequency Dependency of Fatigue Life and Internal Heating of a Fiber-Reinforced Ceramic Matrix Composite," *Journal of American Ceramics Society*, 1994.

## 9. Procedure

9.1 *Test Specimen Dimensions*—Determine the thickness and width of the gage section of each test specimen to within 0.02 mm on at least three different cross-sectional planes in the gage section. To avoid damage in the critical gage section area perform these measurements optically (for example, an optical comparator). Alternatively, mechanical measurements may be made using flat-anvil, ball-tipped, or sharp-anvil micrometers exercising extreme caution to prevent damage to the test specimen gage section. In any case the resolution of the instrument shall be as specified in 6.7. Record and report the measured dimensions and locations of the measurements for use in the calculation of stresses and strains. Use the average of the multiple measurements in the stress calculations.

9.1.1 Conduct periodic, if not 100 %, inspection/measurements of all test specimens and test specimen dimensions to ensure compliance with the drawing specifications. High-resolution optical methods (for example, an optical comparator) or high-resolution digital point contact methods (for example, coordinate measurement machine) are satisfactory as long as the equipment meets the specifications in 6.7.

NOTE 1—The frequency of occurrence of gage section fractures and bending in the gage section are dependent on proper overall test specimen dimensions within the required tolerances.

9.1.2 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 perpendicular to the tensile axis. When quantified, report surface roughness as average surface roughness,  $R_a$ , or root-mean-square surface roughness,  $R_q$ , at a minimum.

### 9.2 Test Modes and Rates:

9.2.1 *General*—Test modes and rates can have distinct and strong influences on the cyclic fatigue behavior of CFCCs even at ambient temperatures depending on test environment or condition of the test specimen. Test modes may involve load, displacement, or strain control. Maximum and minimum test levels as well as frequency and wave form shape will depend on the purpose for which the tests are being conducted. Previous studies have shown decreasing cyclic fatigue life under load control for increasing frequency<sup>10</sup> and decreasing load ratio,  $R$ <sup>11</sup>. Sine waves provide smooth transitions from maximums to minimums.  $R$  ratios of 0.1 are often used for maximum amplitude effect while avoiding a slack (that is, loose and non-tensioned) force train. Frequencies are chosen to reflect service conditions, generally ranging from 1 to 10 Hz for exploratory tests and extending to 1000 Hz for materials characterization for components. In all cases report the test mode, maximum test level, minimum test level, frequency, wave form, and  $R$  or  $A$  ratio.

9.2.2 Prior to cyclic fatigue testing, test a sufficient number of control test specimens in accordance with Test Method C 1275. STP 588<sup>9</sup> may provide guidance for the number of control specimens to test. Use the average of the control tests

to establish the 100 % level (that is, the uniaxial, monotonic tensile strength of the material) of the cyclic fatigue tests. Cyclic fatigue tests can then be conducted at maximum stresses or strains as a percentages of this 100 % level.

### 9.3 Conducting the Cyclic Fatigue Test:

9.3.1 *Mounting the Test Specimen*—Each grip interface and test specimen geometry discussed in Test Method C 1275 will require a unique procedure for mounting the test specimen in the force train. Identify and report any special components which are required for each test. Mark the test specimen with a non corroding, indelible marker as to top and bottom and front (side facing the operator) in relation to the test machine. In the case of strain-gaged specimens, orient the test specimen such that the front of the test specimen and a unique strain gage (for example, strain gage 1 designated SGI) coincide.

9.3.2 *Preparations for Testing*—Set the test mode and frequency on the testing machine. Preload the test specimen to remove the slack from the force train. Determine and report the amount of preload for each situation, specific to each material tensile test specimen geometry. If strain is being measured, either mount the extensometer on the test specimen gage section and zero the output, or, attach the lead wires of the strain gages to the signal conditioner and zero the outputs. If temperature is being measured, attach the temperature recording equipment. If required, ready the autograph data acquisition systems for periodic data logging.

NOTE 2—If strain gages are used to monitor bending, zero the strain gages with the test specimen attached at only one end of the fixtures, that is, hanging free. This will ensure that bending due to the grip closure is factored into the measured bending. In addition, if test specimen self-heating due to hysteresis is anticipated, strain gages should be temperature compensated following accepted practice.

9.3.3 *Conducting the Test*—Initiate the data acquisition. Initiate the test mode. After testing has begun, check the loading often unless the testing machine is equipped with automatic force maintainers to ensure that loads at peaks and valleys do not vary by greater than 1.0 %. Refer to Practice E 467. Mass inertia effects of the machine fixtures and test specimens shall be calibrated by means of strain gages, Wheatstone bridge, and an oscilloscope or oscillograph for the particular load range and machine speed being used. Corrections of loading shall be made to offset these effects and produce the desired loading cycle. Refer to Practice E 467.

9.3.4 Record the number of cycles and corresponding test conditions at the completion of testing. A test may be terminated for one of several conditions: test specimen fracture; reaching a pre-determined number of run-out cycles; reaching a pre-determined test specimen compliance or material elastic modulus, reaching a pre-determined phase lag between control mode and response. At test termination, disable the action of the test machine and the data collection of the data acquisition system. Carefully remove the test specimen from the grip interfaces. Take care not to damage the fracture surfaces, if they exist, by preventing them from contact with each other or other objects. Place the test specimen along with any fragments from the gage section into a suitable, non-metallic container for later analysis.

9.3.5 Determine and report the test temperature and relative

<sup>11</sup> J.W. Holmes, "Influence of Stress-Ratio on the Elevated Temperature Fatigue of a SiC Fiber-Reinforced Si<sub>3</sub>N<sub>4</sub> Composite," *J. Am. Ceram. Soc.* **74**, (7), 1639-45, 1991.

humidity in accordance with Test Method E 337 at a minimum at the beginning and end of each test, and hourly if the test duration is greater than 1 h.

9.3.6 *Post-Test Dimensions*—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 test specimen as tested (and marked) and negative (–) measurements toward the bottom of the test specimen as tested (and marked).

9.3.6.1 Note that results from test specimens fracturing outside the uniformly stressed gage section may be considered anomalous. Results from test specimens fracturing outside the gage section can still be used as censored tests (that is, tests in which a stress at least equal to that calculated by Eq 4 was sustained in the uniform gage section before the test was prematurely terminated by a nongage section fracture). Censored tests are discussed in STP 91A.<sup>8</sup> To complete a required statistical sample for purposes of establishing cyclic fatigue behaviour without censoring, test one replacement specimen for each test specimen which fractures outside the gage section.

9.4 *Fractography*—Conduct visual examination and light microscopy to determine the mode and type of fracture (that is, brittle or fibrous). In addition, although quantitatively beyond the scope of this practice, 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*—The basic formulae for calculating engineers parameters are given as follows. Additional guidelines for interpretation and reporting cyclic fatigue results are contained in STP 91A,<sup>8</sup> STP 588<sup>9</sup>, and Practice E 739.

10.2 *Engineering Stress*—Calculate the engineering stress as:

$$\sigma = \frac{P}{A} \quad (4)$$

where:

$\sigma$  = engineering stress, MPa,

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

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

The cross-sectional area  $A$  is calculated as:

$$A = wb \quad (5)$$

where  $w$  and  $b$  are the average width and average thickness of the gage section, respectively, mm, as detailed in 9.1.

10.3 *Engineering Strain*—Calculate the engineering strain as:

$$\epsilon = \frac{(l - l_0)}{l_0} \quad (6)$$

where:

$\epsilon$  = engineering strain,

$l$  = extensometer gage length at any time, mm, and

$l_0$  = original gage length of the extensometer in units of mm.

In the case of strain gages, strain is measured directly and Eq 6 is not required.

10.4 *Tensile Strength*—Calculate the tensile strength as:

$$S_u = \frac{P_{max}}{A} \quad (7)$$

where:

$S_u$  = tensile strength, MPa, and

$P_{max}$  = maximum force, N

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

$$E = \frac{\Delta\sigma}{\Delta\epsilon} \quad (8)$$

where:

$E$  = the modulus of elasticity, and

$\Delta\sigma/\Delta\epsilon$  the slope of the  $\sigma - \epsilon$  curve within the linear region as discussed in 10.8 of Test Method C 1275. Note that the modulus of elasticity may not be defined for materials which exhibit entirely non-linear  $\sigma - \epsilon$  curves.

## 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 practice:

11.1.1 Date and location of testing,

11.1.2 Tensile test specimen geometry used (include engineering drawing). For end-tabbed test 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 and configuration 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 Number ( $n$ ) of test specimens tested validly (for example, fracture in the gage section). In addition, report the total of number of test specimens tested ( $n_T$ ) to provide an indication of the expected success rate of the particular test specimen geometry and test apparatus,

11.1.8 Where feasible and possible, all relevant material data including vintage or billet identification. As a minimum, report the date the material was manufactured,

11.1.8.1 For commercial materials, where feasible and possible, report the commercial designation and lot number. As a minimum include a short description of reinforcement (type, layup, etc.), fiber volume fraction, and bulk density,

11.1.8.2 For non-commercial materials, where feasible and possible, report the major constituents and proportions as well as the primary processing route including green state and consolidation routes. Also report fiber volume fraction, matrix porosity, and bulk density. Fully 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.),

11.1.9 Description of the method of test specimen preparation including all stages of machining, cleaning, and storage time and method before testing,

11.1.10 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 test specimen,

11.1.11 Test environment and intervals at which measured, including relative humidity (Test Method E 337), ambient temperature, and atmosphere (for example, ambient air, dry nitrogen, silicone oil, etc.),

11.1.12 Test mode (load, displacement, or strain control), wave form, actual frequency of testing, and  $R$  or  $A$  ratio,

11.1.13 The stress-life or strain-life developed for the entire test series Practice E 468 and E 739. Alternatively or additionally, stress-time or strain-time can be developed for the entire test series,

11.1.14 Percent bending and corresponding average strain in the specimen recorded during the verification as measured at the beginning and end of the test series. In addition, plot a curve of percent bending vs. the test parameter (load, displacement, strain, etc.) to assist in understanding the role of bending over the course of testing from the minimum to the maximum, and

11.1.15 Mean, standard deviation, and coefficient of variation for the following measured properties of the control specimens for each test series as determined using Test Method C 1275:

11.1.15.1 Tensile strength,  $S_u$

11.1.15.2 Strain at tensile strength,  $\epsilon_u$ ,

11.1.15.3 Fracture strength,  $S_f$ ,

11.1.15.4 Strain at fracture strength,  $\epsilon_f$ ,

11.1.15.5 Modulus of elasticity,  $E$ , (if applicable),

11.1.15.6 Proportional limit stress,  $\sigma_o$  (if applicable) and method of determination,

11.1.15.7 Strain at proportional limit stress,  $\epsilon_o$  (if applicable),

11.1.15.8 Modulus of resilience,  $U_R$  (if applicable), and

11.1.15.9 Modulus of toughness,  $U_T$  (if applicable).

11.2 *Individual Test Specimens*—Report the following information for each test specimen tested. Note and report any significant deviations from the procedures and requirements of this practice.

11.2.1 Pertinent overall specimen dimensions, if measured, such as total length, length of gage section, gripped section dimensions, etc, mm,

11.2.2 Average surface roughness,  $\mu\text{m}$ , if measured, of gage section and the direction of measurement,

11.2.3 Average cross-sectional dimensions, if measured, or cross-sectional dimensions at the plane of fracture in mm,

11.2.4 Plots of periodic stress-strain curves, if so recorded, and corresponding number of cycles,

11.2.5 Maximum cyclic stress, strain, or displacement,

11.2.6 Minimum cyclic stress, strain, or displacement,

11.2.7 Amplitude of cyclic stress, strain, or displacement,

11.2.8  $R$  or  $A$  ratio,

11.2.9 Wave form and frequency of testing, including any hold times,

11.2.10 Cycles or time to test termination, or both, and criterion for test termination,

11.2.11 Fracture location relative to the gage section midpoint in units of mm (+ is toward the top of the test specimen as marked and– is toward the bottom of the specimen as marked with 0 being the gage section midpoint) if relevant, and

11.2.12 Appearance of test specimen after fracture as suggested in 9.4.

## 12. Keywords

12.1 ceramic matrix composite; CFCC; continuous fiber ceramic composite; cyclic fatigue; S-N curve; tension-tension cyclic fatigue

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