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Standard Test Method for Determination of Slow Crack Growth Parameters of Advanced Ceramics by Constant Stress-Rate Flexural Testing at Elevated Temperatures[1]

This standard is issued under the fixed designation C 1465; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of slow crack growth (SCG) parameters of advanced ceramics by using constant stress-rate flexural testing in which flexural strength is determined as a function of applied stress rate in a given environment at elevated temperatures. The strength degradation exhibited with decreasing applied stress rate in a specified environment is the basis of this test method which enables the evaluation of slow crack growth parameters of a material.

NOTE 1—This test method is frequently referred to as "dynamic fatigue" testing (Refs **(1-3)**)[2] in which the term "fatigue" is used interchangeably with the term "slow crack growth." To avoid possible confusion with the "fatigue" phenomenon of a material which occurs exclusively under cyclic loading, as defined in Terminology E 1823, this test method uses the term "constant stress-rate testing" rather than "dynamic fatigue" testing.

NOTE 2—In glass and ceramics technology, static tests of considerable duration are called "static fatigue" tests, a type of test designated as stress-rupture (Terminology E 1823).

1.2 This test method is intended primarily to be used for *negligible* creep of test specimens, with specific limits on creep imposed in this test method.

1.3 This test method applies primarily to advanced ceramics that are macroscopically homogeneous and isotropic. This test method may also be applied to certain whisker- or particle-reinforced ceramics that exhibit macroscopically homogeneous behavior.

1.4 This test method is intended for use with various test environments such as air, vacuum, inert, and any other gaseous environments.

1.5 Values expressed in this standard test are in accordance with the International System of Units (SI) and Practice E 380.

1.6 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 1145 Terminology of Advanced Ceramics[3]

C 1161 Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature[3]

C 1211 Test Method for Flexural Strength of Advanced Ceramics at Elevated Temperatures[3]

C 1239 Practice for Reporting Uniaxial Strength Data and Estimating Weibull Distribution Parameters for Advanced Ceramics[3]

C 1322 Practice for Fractography and Characterization of Fracture Origins in Advanced Ceramics[3]

C 1368 Test Method for Determination of Slow Crack Growth Parameters of Advanced Ceramics by Constant Stress-Rate Flexural Testing at Ambient Temperature[3]

E 4 Practices for Force Verification of Testing Machines[4]

E 6 Terminology Relating to Methods of Mechanical Testing[4]

E 220 Test Method for Calibration of Thermocouples by Comparison Techniques[5]

E 230 Specification and Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples[5]

E 337 Test Method for Measured Humidity with a Psychrometer (the Measurement of Wet- and Dry-Bulb Temperatures)[6]

E 380 Practice for Use of International System of Units (SI) (The Modernized Metric System)[7]

E 1823 Terminology Relating to Fatigue and Fracture Testing[4]

3. Terminology

3.1 *Definitions*—The terms described in Terminologies C 1145, E 6, and E 1823 are applicable to this test method. Specific terms relevant to this test method are as follows:

3.1.1 *advanced ceramic, n*—a highly engineered, high-performance, predominately, nonmetallic, inorganic, ceramic material having specific functional attributes. **(C 1145)**

[1] This test method is under the jurisdiction of ASTM Committee C-28 on Advanced Ceramics and is the direct responsibility of Subcommittee C28.01 on Properties and Performance.

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[2] The boldface numbers in parentheses refer to the list of references at the end of this standard.

[3] *Annual Book of ASTM Standards*, Vol 15.01.

[4] *Annual Book of ASTM Standards*, Vol 03.01.

[5] *Annual Book of ASTM Standards*, Vol 14.03.

[6] *Annual Book of ASTM Standards*, Vol 11.03.

[7] *Annual Book of ASTM Standards*, Vol 14.02.

3.1.2 *constant stress rate, $\dot{\sigma}$* [$FL^{-2}\,t^{-1}$], *n*—a constant rate of increase of maximum flexural stress applied to a specified beam by using either a constant load or constant displacement rate of a testing machine.

3.1.3 *environment, n*—the aggregate of chemical species and energy that surrounds a test specimen. **(E 1150)**

3.1.4 *environmental chamber, n*—a container surrounding the test specimen and capable of providing controlled local environmental condition.

3.1.5 *flexural strength,* σ_f [FL^{-2}], *n*—a measure of the ultimate strength of a specified beam specimen in bending determined at a given stress rate in a particular environment.

3.1.6 *flexural strength-stress rate diagram*—a plot of flexural strength as a function of stress rate. Flexural strength and stress rate are both plotted on logarithmic scales.

3.1.7 *flexural strength-stress rate curve*—a curve fitted to the values of flexural strength at each of several stress rates, based on the relationship between flexural strength and stress rate:

$$\log \sigma_f = [1/(n + 1)] \log \dot{\sigma} + \log D \text{ (see Appendix X1)}$$

3.1.7.1 *Discussion*—In the ceramics literature, this is often called a "dynamic fatigue" curve.

3.1.8 *fracture toughness,* K_{IC} [$FL^{-3/2}$], *n*—a generic term for measures of resistance to extension of a crack. **(E 616)**

3.1.9 *inert flexural strength* [FL^{-2}], *n*—a measure of the strength of a specified beam specimen in bending as determined in an appropriate inert condition whereby no slow crack growth occurs.

3.1.9.1 *Discussion*—An inert condition at near room temperature may be obtained by using vacuum, low temperatures, very fast test rates, or any inert media. However, at elevated temperatures, the definition or concept of an inert condition is unclear since temperature itself acts as a degrading environment. It has been shown that for some ceramics one approach to obtain an inert condition (thus, inert strength) at elevated temperatures is to use very fast (ultra-fast) test rates $\geq 3 \times 10^4$ MPa/s, where the time for slow crack growth would be minimized or eliminated **(4)**.

3.1.10 *slow crack growth* (SCG), *n*—subcritical crack growth (extension) which may result from, but is not restricted to, such mechanisms as environmentally assisted stress corrosion or diffusive crack growth.

3.1.11 *stress intensity factor,* K_I [$FL^{-3/2}$], *n*—the magnitude of the ideal-crack-tip stress field (stress-field singularly) subjected to Mode I loading in a homogeneous, linear elastic body. **(E 616)**

3.1.12 *R-curve, n*—a plot of crack-extension resistance as a function of stable crack extension. **(E 616)**

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *slow crack growth parameters, n and D, n*—the parameters estimated as constants in the flexural strength (in megapascals)-stress rate (in megapascals per second) equation, which represent a measure of susceptibility to slow crack growth of a material (see Appendix X1). For the units of *D*, see 9.3.1.

4. Significance and Use

4.1 For many structural ceramic components in service, their use is often limited by lifetimes that are controlled by a process of slow crack growth. This test method provides the empirical parameters for appraising the relative slow crack growth susceptibility of ceramic materials under specified environments at elevated temperatures. This test method is similar to Test Method C 1368 with the exception that provisions for testing at elevated temperatures are given. Furthermore, this test method may establish the influences of processing variables and composition on slow crack growth as well as on strength behavior of newly developed or existing materials, thus allowing tailoring and optimizing material processing for further modification. In summary, this test method may be used for material development, quality control, characterization, and limited design data generation purposes.

NOTE 3—Data generated by this test method do not necessarily correspond to crack velocities that may be encountered in service conditions. The use of data generated by this test method for design purposes may entail considerable extrapolation and loss of accuracy.

4.2 In this test method, the flexural stress computation is based on simple beam theory, with the assumptions that the material is isotropic and homogeneous, the moduli of elasticity in tension and compression are identical, and the material is linearly elastic. The average grain size should be no greater than one fiftieth (1/50) of the beam thickness.

4.3 In this test method, the test specimen sizes and test fixtures were chosen in accordance with Test Method C 1211, which provides a balance between practical configurations and resulting errors, as discussed in Refs **(5, 6)**. Only the four-point test configuration is used in this test method.

4.4 In this test method, the slow crack growth parameters (*n* and *D*) are determined based on the mathematical relationship between flexural strength and applied stress rate, $\log \sigma_f = [1/(n + 1)] \log \dot{\sigma} + \log D$, together with the measured experimental data. The basic underlying assumption on the derivation of this relationship is that slow crack growth is governed by an empirical power-law crack velocity, $v = A[K_I/K_{IC}]^n$ (see Appendix X1).

NOTE 4—There are various other forms of crack velocity laws which are usually more complex or less convenient mathematically, or both, but may be physically more realistic **(7)**. The mathematical analysis in this test method does not cover such alternative crack velocity formulations.

4.5 In this test method, the mathematical relationship between flexural strength and stress rate was derived based on the assumption that the slow crack growth parameter is at least $n \geq 5$ **(1, 8)**. Therefore, if a material exhibits a very high susceptibility to slow crack growth, that is, $n < 5$, special care should be taken when interpreting the results.

4.6 The mathematical analysis of test results according to the method in 4.4 assumes that the material displays no rising *R*-curve behavior, that is, no increasing fracture resistance (or crack-extension resistance) with increasing crack length. It should be noted that the existence of such behavior cannot be determined from this test method. The analysis further assumes that the same flaw types control strength over the entire test range. That is, no new flaws are created, and the flaws that control the strength at the highest stress rate control the strength at the lowest stress rate.

4.7 Slow crack growth behavior of ceramic materials can vary as a function of mechanical, material, thermal, and

environmental variables. Therefore, it is essential that test results accurately reflect the effects of specific variables under study. Only then can data be compared from one investigation to another on a valid basis, or serve as a valid basis for characterizing materials and assessing structural behavior.

4.8 The strength of advanced ceramics is probabilistic in nature. Therefore, slow crack growth that is determined from the flexural strengths of a ceramic material is also a probabilistic phenomenon. Hence, a proper range and number of test rates in conjunction with an appropriate number of specimens at each test rate are required for statistical reproducibility and design **(2)**. Guidance is provided in this test method.

NOTE 5—For a given ceramic material/environment system, the SCG parameter *n* is independent of specimen size although its reproducibility is dependent on the variables previously mentioned. By contrast, the SCG parameter *D* depends significantly on strength, and thus on specimen size (see Eq X1.7).

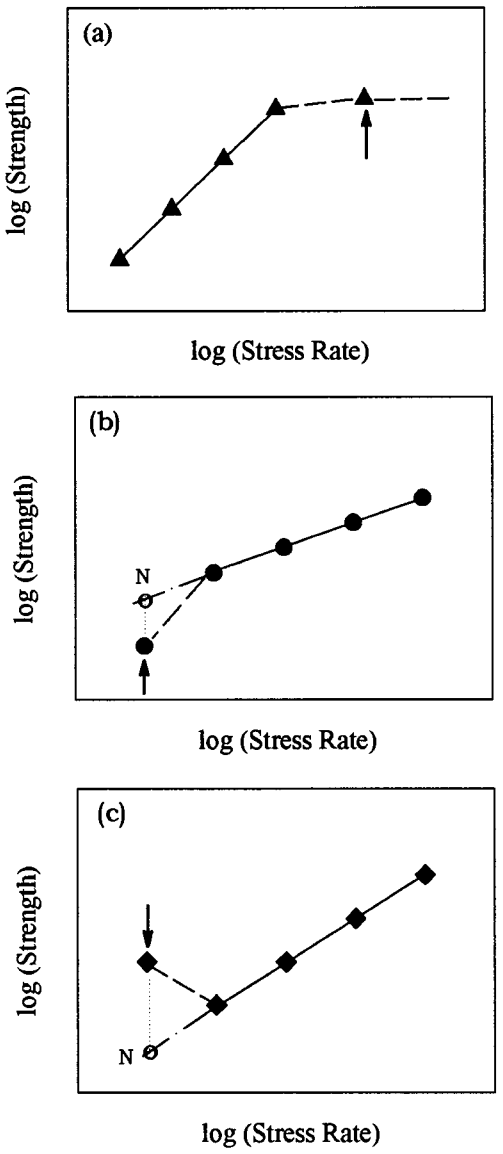
4.9 The elevated-temperature strength of a ceramic material for a given test specimen and test fixture configuration is dependent on its inherent resistance to fracture, the presence of flaws, test rate, and environmental effects. Analysis of a fracture surface, fractography, though beyond the scope of this test method, is highly recommended for all purposes, especially to verify the mechanism(s) associated with failure (refer to Practice C 1322).

5. Interferences

5.1 Slow crack growth may be the product of both mechanical and chemical driving forces. The chemical driving force for a given material can strongly vary with the composition and temperature of a test environment. Note that slow crack growth testing is time-consuming. It may take several weeks to complete testing of a typical, advanced ceramic. Because of this long test time, the chemical variables of the test environment must be prevented from changing throughout the tests. Inadequate control of these chemical variables may result in inaccurate strength data and SCG parameters, especially for materials that are sensitive to the environment.

5.2 Significant creep at both higher temperatures and lower test rates results in nonlinearity in stress-strain relations as well as accumulated tensile damage in flexure **(9)**. This, depending on the degree of nonlinearity, may limit the applicability of linear elastic fracture mechanics (LEFM), since the resulting relationship between strength and stress rate derived under constant stress-rate testing condition is based on an LEFM approach with negligible creep (creep strain less than 0.1 %). Therefore, creep should be kept as minimal as possible, as compared to the total strain at failure (see 8.11.2).

5.3 Depending on the degree of SCG susceptibility of a material, the linear relationship between log (*flexural strength*) and log (*applied stress rate*) (see Appendix X1) may start to deviate at a certain high stress rate, at which slow crack growth diminishes or is minimized due to the extremely short test duration. Strengths obtained at higher stress rates (>1000 MPa/s) may remain unchanged so that a plateau is observed in the plot of strength versus stress rate, see Fig. 1a **(4)**. If the strength data determined in this plateau region are included in the analysis, a misleading estimate of the SCG parameters will be obtained. Therefore, the strength data in the plateau shall be



NOTE 1—The arrows indicate unacceptable data points. The data point marked with 'N', in which a significant nonlinearity occurs, indicates a strength value estimated by extrapolation of the linear regression line represented by the rest of the strength data.

FIG. 1 Schematic Diagrams Showing Unacceptable Data Points in Constant Stress-Rate Testing at Elevated Temperatures

excluded as data points in estimating the SCG parameters of the material. This test method addresses this issue by recommending that the highest stress rate be ≤1000 MPa/s.

5.4 A considerable strength degradation may be observed at lower stress rates and higher temperatures for some materials. In these cases, excessive creep damage in the form of creep cavities, micro- or macro-cracks, or both, develop in the tensile surface **(10-13)**. This results in a nonlinearity in the relationship between log (*flexural strength*) and log (*applied stress rate*), see Fig. 1b. It has been reported that the strength degradation with respect to the expected normal strength (at Point N in Fig. 1b) ranged from 15 to 50 % **(10-12)**. If these data points are used in the analysis, then an underestimate of the SCG parameters will be obtained. Hence, the strength data

exhibiting such a significant strength degradation occurring at lower stress rates shall be excluded as data points in obtaining the SCG parameters of the material.

5.5 Contrary to the case of significant strength degradation, an appreciable strength increase may occur for some ceramics at lower stress rates (see Fig. 1c), due to crack healing or crack tip blunting which dominates slow crack growth **(10, 14)**. It has been reported that the strength increase with respect to the expected normal strength (at point N in Fig. 1c) ranged from 15 to 60 % **(10, 14)**. Since the phenomenon results in a deviation from the linear relationship between log (*flexural strength*) and log (*applied stress rate*), an overestimate of SCG parameters may be obtained if such strength data are included in the analysis. Therefore, any data exhibiting a significant or obvious increase in strength at lower stress rates shall be excluded as data points in estimating the SCG parameters of the material.

NOTE 6—It has been shown that some preloading (up to 80 % of fracture load) prior to testing may be used to minimize or eliminate the strength-increase phenomenon by minimizing or eliminating a chance for crack healing (or blunting) through shortening test time, as verified on some advanced ceramics such as alumina and silicon nitride **(10, 15)**. In general, preloading may be effective to reduce overall creep deformation of test specimens due to reduced test time. Refer to 8.10 for more information regarding preloading and its application.

5.6 Surface preparation of test specimens can introduce fabrication flaws that may have pronounced effects on flexural strength. Machining damage imposed during specimen preparation can be either a random interfering factor, or an inherent part of the strength characteristics to be measured. Surface preparation can also lead to residual stress. Universal or standardized test methods of surface preparation do not exist. It should be understood that the final machining steps may or may not negate machining damage introduced during the early coarse or intermediate machining steps. In some cases, specimens need to be tested in the as-processed condition to simulate a specific service condition. Therefore, specimen fabrication history may play an important role in strength behavior, which consequently may affect the values of the SCG parameters to be determined.

6. Apparatus

6.1 *Test Machine*—Test machines used for this test method shall conform to the requirements of Practices E 4. Test specimens may be loaded in any suitable test machine provided that uniform test rates, either using load-control or displacement-control mode, can be maintained. The loads used in determining flexural strength shall be accurate within ±1.0 % at any load within the selected test rate and load range of the test machine as defined in Practices E 4. The test machine shall have a minimum capability of applying at least four test rates with at least three orders of magnitude, ranging from 10^{-1} to 10^{-2} N/s for load-control mode, and from 10^{-7} to 10^{-4} m/s for displacement-control mode.

6.2 *Test Fixtures*—The configurations and mechanical properties of test fixtures shall be in accordance with Test Method C 1211. The materials from which the test fixtures, including bearing cylinders, are fabricated shall be effectively inert to the test environment so that they do not significantly react with or contaminate either the test specimen or the test environment. In addition, the test fixtures must remain elastic under test conditions (load and temperature).

NOTE 7—Various grades of silicon carbide (such as hot-pressed or sintered) and high-purity aluminas are candidate materials for test fixtures as well as load train. The load-train material should also be effectively inert to the test environment and remain elastic under test conditions. For more specific information regarding use of appropriate materials for fixtures and load train with respect to test temperatures, refer to Section 6 of Test Method C 1211.

6.2.1 *Four-Point Flexure*—The four-point ¼-point fixture configuration (see Fig. 2) as described in Test Method C 1211 shall be used in this test method. The nominal outer (support) span (L) for each test fixture is L = 20 mm, 40 mm, and 80 mm, respectively, for A, B, and C test fixtures. The use of three-point flexure is excluded from this test method.

6.2.2 *Bearing Cylinders*—The requirements of dimensions and mechanical properties of bearing cylinders as described in Test Method C 1211 shall be used in this test method. The bearing cylinders shall be free to rotate in order to relieve frictional constraints, as described in Test Method C 1211.

6.2.3 *Semiarticulating Four-Point Fixture*—The semiarticulating four-point fixture as described in Test Method C 1211 may be used in this test method. This fixture shall be used when the parallelism requirements of test specimens are met in accordance with Test Method C 1211.

6.2.4 *Fully Articulating Four-Point Fixture*—The fully articulating four-point fixture as described in Test Method C 1211 may be used in this test method. Specimens that do not meet the parallelism requirements in Test Method C 1211, due to the nature of fabrication process (as-fired, heat-treated, or oxidized), shall be tested in this fully articulating fixture.

6.3 *System Compliance*—The test fixture and load train shall be sufficiently stiff so that at least 80 % of the crosshead or actuator movement of the test machine is imposed onto the test specimen up to the point of fracture. The test fixture and load train shall not undergo creep or nonlinear deformation under either load or displacement control.

NOTE 8—Compliance of the test fixture and load train at the test

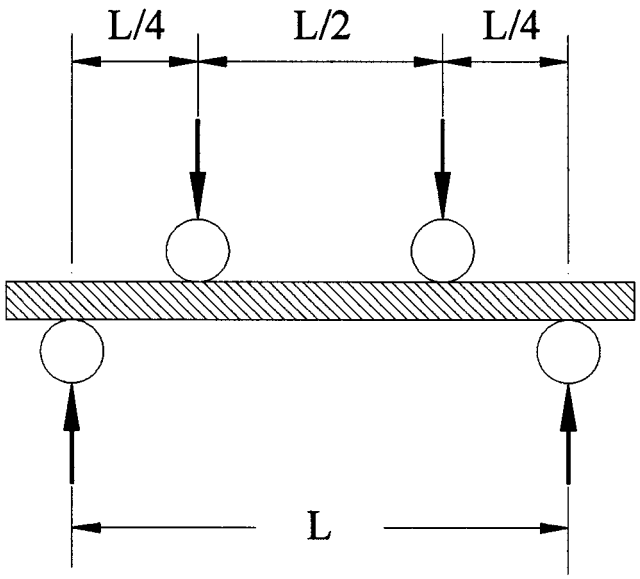


FIG. 2 Four-Point-¼ Point Flexural Test Fixture Configuration

temperature can be estimated by inserting a rigid block of a ceramic material onto the test fixture with the loading bearing cylinders in place, and loading it to the maximum anticipated fracture load while recording a load-deflection curve. The compliance corresponds to the inverse of the slope of the load-deflection curve. It is recommended that the block be at least five times thicker than the test specimen depth and one to two times wider than the test specimen width. Any other block whose rigidity (equal to the inverse of compliance) is greater than at least 120 times that of the test specimen can be used provided that it can fit the test fixture. A typical test machine equipped with common load train and test fixtures shows that more than 90 % of the total compliance stems from the test specimen itself, so that more than 90 % of crosshead or actuator movement of test machine can be imposed on the test specimen.

6.4 *Heating Apparatus*—The heating systems such as furnace, temperature measuring device and thermocouple shall conform to the requirements as described in Test Method C 1211.

6.4.1 *Furnace and Temperature Readout Device*—The furnace shall be capable of maintaining the test specimen temperature within ±2°C during each testing period. The temperature readout device shall have a resolution of 1°C or lower. The furnace system shall be such that thermal gradients are minimal in the test specimen so that no more than a 5°C differential exists from end-to-end in the test specimen.

6.4.2 *Thermocouples*:

6.4.2.1 The specimen temperature shall be monitored by a thermocouple with its tip situated no more than 1 mm from the midpoint of the test specimen. Either a fully sheathed or exposed bead junction may be used. If a sheathed tip is used, it must be verified that there is negligible error associated with the covering.

NOTE 9—Exposed thermocouple beads have greater sensitivity, but they may be exposed to vapors that can react with the thermocouple materials. (For example, silica vapors will react with platinum.) Beware of the use of heavy-gage thermocouple wire, thermal gradients along the thermocouple length, or excessively heavy-walled insulators, all of which can lead to erroneous temperature readings.

NOTE 10—The thermocouple tip may contact the test specimen, but only if there is certainty that thermocouple tip or sheathing material will not interact chemically with the test specimen. Thermocouples may be prone to breakage if they are in contact with the test specimen.

6.4.2.2 A separate thermocouple may be used to control the furnace if necessary, but the test specimen temperature shall be the reported temperature of the test.

NOTE 11—Tests are sometimes conducted in furnaces that have thermal gradients. The small size of test specimens will alleviate thermal gradient problems, but it is essential to monitor the temperature at the test specimen.

6.4.2.3 The thermocouple(s) shall be calibrated in accordance with Test Method E 220 and Specification and Tables E 230. The thermocouples shall be periodically checked since calibration may drift with usage or contamination.

6.4.2.4 The measurement of temperature shall be accurate to within ±5°C. The accuracy shall include the error inherent to the thermocouple as well as any errors in the measuring instruments.

NOTE 12—Resolution should not be confused with accuracy. Beware of recording instruments that read out to 1°C (resolution) but have an accuracy of only ±10°C or ±½ % of full-scale (for example, ½ % of 1200°C is 6°C).

NOTE 13—Temperature measuring instruments typically approximate the temperature-electromotive force (EMF, in millivolt) tables, and may have an error of a few degrees.

6.4.2.5 The appropriate thermocouple extension wire should be used to connect a thermocouple to the furnace controller and temperature readout device, which shall have either a cold junction or a room-temperature compensation circuit. Special care should be directed toward connecting the extension wire with the correct polarity.

6.5 *Environmental Facility*—The furnace may have an air, inert, vacuum, or any other gaseous environment, as required. If testing is conducted in any gaseous environment other than ambient air, an appropriate environmental chamber shall be constructed to facilitate handling and monitoring of the test environment so that constant test conditions can be maintained. The chamber shall be effectively corrosion-resistant to the test environment so that it does not react with or change the environment. If it is necessary to direct load through bellows, fittings, or seal, it shall be verified that load losses or errors do not exceed 1 % of the prospective failure loads.

6.6 *Deflection Measurement*—When determined, measure deflection of the test specimen close to the midpoint or inner load point(s) (tension side). The method to measure the deflection of the midpoint relative to the two inner load points (for example, three-probe extensometer) can also be utilized, if determined. The deflection-measuring equipment shall be capable of resolving 1×10^{-3} mm. Deflection measurement of test specimens is particularly important at the test conditions of lower test rates or higher test temperatures, or both, and is highly recommended to ensure that creep strain of test specimens is within the allowable limit (see 8.11.2).

NOTE 14—Alternatively, crosshead or actuator displacement may be used to infer deflection of the test specimen. However, care should be taken in interpreting the result since crosshead or actuator displacement generally may not be as sensitive as measurements taken on the specimen itself.

NOTE 15—When a contact-type deflection-measuring equipment such as LVDT is employed, it is important not to damage the contact area of specimens due to prolonged contact with the deflection-measuring probe, particularly at lower test rates and higher test temperatures. Any spurious damage may act as a failure-originating source so that the contacting force should be kept minimal, in the range from 0.5 to 2 N. A general guideline is that the maximum contacting force is dependent on specimen size such that 0.5 N for Size A, 1 N for Size B, and 2 N for Size C specimen. The probe with its tip rounded may be fabricated with the same material as test specimens or with sintered silicon carbide.

6.7 *Data Acquisition*—Accurate determination of both fracture load and test time is important since they affect not only fracture strength but applied stress rate. At the minimum, an autographic record of applied load versus time should be determined during testing. Either analog chart recorders or digital data acquisition systems can be used for this purpose. An analog chart recorder should be used in conjunction with the digital data acquisition system to provide an immediate record of the test as a supplement to the digital record. Recording devices shall be accurate to 1.0 % of the recording range and should have a minimum data acquisition rate of 1 kHz with a response of 5 kHz or greater deemed more than sufficient. The appropriate data acquisition rate depends on the

test rate: The greater the test rate, the greater the acquisition rate; and vice versa.

7. Test Specimen

7.1 *Specimen Size*—The types and dimensions of rectangular beam specimens as described in Test Method C 1211 shall be used in this test method. The nominal dimensions of each type of test specimens are 2.0 by 1.5 by 25 mm (minimum), respectively, in width (*b*), depth (*d*), and length for Size A test specimens; 4.0 by 3.0 by 45 mm (minimum) for Size B test specimens; and 8.0 by 6.0 by 90 mm (minimum) for Size C test specimens.

7.2 *Specimen Preparation*—Specimen fabrication and preparation methods as described in Test Method C 1211 shall be used in this test method.

7.3 *Specimen Dimensions*—If there is a concern about a dimensional change in test specimens by possible reaction/reaction products due to a prolonged test duration particularly at very low test rates, measure test specimen dimensions prior to testing. Determine the thickness and width of each test specimen to within 0.002 mm either optically or mechanically using a flat, anvil-type micrometer. Exercise extreme caution to prevent damage to the critical area of the test specimen. Otherwise, measure the test specimen dimensions after testing (see 8.12.2)

7.4 *Handling and Cleaning*—Exercise care in handling and storing specimens in order to avoid introducing random and severe flaws, which might occur if the specimens were allowed to impact or scratch each other. If desired or necessary, clean test specimens with an appropriate cleaning medium such as methanol, high-purity (>99 %) isopropyl alcohol, or any other cleaning agent, since surface contamination of test specimens by lubricant, residues, rust, or dirt might affect slow crack growth for certain test environments. Also, residue from the cleaning medium, if any, shall not have any undesirable effect on slow crack growth (strength) of test specimens.

7.5 *Number of Test Specimens*—The required number of test specimens depends on the statistical reproducibility of SCG parameters (*n* and *D*) to be determined. The statistical reproducibility is a function of strength scatter (Weibull modulus), number of test rates, range of test rates, and SCG parameter (*n*). Because of these various variables, there is no single guideline as to the determination of the appropriate number of test specimens. A minimum of 10 specimens per test rate is recommended in this test method. The total number of test specimens shall be at least 40, with at least four different test rates (see 8.2.2). The number of test specimens (and test rates) recommended in this test method has been established with the intent of determining reasonable confidence limits on both strength distribution and SCG parameters.

NOTE 16—Refer to Ref **(2)** when a specific purpose is sought for the statistical reproducibility of SCG parameters.

7.6 *Valid Tests*—A valid individual test is one which meets all the following requirements: (*1*) all the test requirements of this test method and (*2*) fracture occurring in the uniformly stressed section (that is, in the inner span) (see 8.12.3).

7.7 *Randomization of Test Specimens*—Since a somewhat large number of test specimens (a minimum of 40) with at least four different test rates is used in this test method, it is highly recommended that all the test specimens provided be randomized prior to testing in order to reduce any systematic error associated with material fabrication or specimen preparation, or both. Randomize the test specimens (using, for example, a random number generator) in groups equal to the number of test rates to be employed, if desired.

8. Procedure

8.1 *Test Fixtures*—Choose the appropriate fixture in the specific test configurations, as described in 6.2. Use the four-point A fixture for the Size A specimens. Similarly, use the four-point B fixture for Size B specimens, and the four-point C fixture for Size C specimens. A fully articulating fixture is required if the specimen parallelism requirements cannot be met.

8.2 *Test Rates*:

8.2.1 The choice of range and number of test rates not only affects the statistical reproducibility of SCG parameters but depends on the capability of a test machine. Since various types of test machines are currently available, no simple guideline regarding the range of test rates can be made. However, when the lower limits of the test rates of most commercial test machines are considered (often attributed to insufficient resolution of crosshead or actuator movement control), it is generally recommended that the lowest test rates be $\geq 10^{-2}$ N/s and 10^{-8} m/s, respectively, for load- and displacement-controlled modes. Choice of the upper limits of the test rates of test machines is dependent on several factors associated with the dynamic response of the crosshead or actuator, the load cell, and the data acquisition system (including the chart recorder, if used). Since these factors vary widely from one test machine to another, depending on their capability, no specific upper limit can be established. However, based on the factors common to many test machines and in order to avoid data generation in a plateau region (see 5.2), it is generally recommended that the upper test rates be $\leq 10^{3}$ N/s and 10^{-3} m/s, respectively, for load- and displacement-control modes.

8.2.2 For a test machine equipped with load-control mode, choose at least four load rates (evenly spaced in a logarithmic scale) covering three orders of magnitude (for example, 10^{-1}, 10^{0}, 10^{1}, and 10^{2} N/s). Similarly, for a test machine equipped with displacement-control mode, choose at least four displacement rates (evenly spaced in a logarithmic scale) covering three orders of magnitude (for example, 10^{-7}, 10^{-6}, 10^{-5}, and 10^{-4} m/s). The use of five or more test rates (evenly spaced in a logarithmic scale) covering four or more orders of magnitude is also allowed if the testing machine is capable and the test specimens are available. In general, the load-control mode provides a better output wave-form than the displacement-control mode, particularly at low test rates. In addition, the specified applied load rate can be directly related to stress rate, regardless of compliance of test frame, load train, fixture and specimen, thus simplifying data analysis. In the displacement-control mode, however, the load rate to be determined is a function of both applied displacement rate and system compliance so that the actual load rate should always be measured and used to calculate a corresponding stress rate, thus making data

analysis complex. Therefore, use of load-control mode is highly recommended.

NOTE 17—When using faster test rates, care must be exercised particularly for the conventional, older electromechanical testing machines equipped with slow-response load cells and chart recorders. In general, such systems have an upper limit stress rate of about 100 MPa/s since the chart recorder and/or the load cell cannot follow load rate and hence cannot correctly monitor the fracture load **(16, 17)**. This factor should be taken into account when the fast crosshead speeds are selected on older testing machines. The minimum time to failure in this case should be within a few seconds (≥3 s). However, the use of a better load cell (for example, piezoelectric load cell) or a fast-response chart recorder or a digital data acquisition system, or both, can improve the existing performance so that higher test rates (up to 2000 MPa/s **(16)** can be achieved. It has been shown that digitally controlled, modern testing machine is capable of applying stress rates up to 1×10^5 MPa/s **(4)**.

8.3 *Assembling Test Fixture/Specimen*:

8.3.1 Examine the bearing cylinders to make sure that they are undamaged, and that there are no reaction products or oxidation that could result in uneven line loading of the test specimen or prevent the bearing cylinders from rolling. Remove and clean, or replace the bearing cylinders, if necessary. Avoid any undesirable dimensional changes in the bearing cylinders, for example, by inadvertently forming a small flat on the cylinder surface when certain abrasion (for example, abrasive paper) is used to remove the reaction products from the cylinders. The same care should be directed toward the contact surfaces in the loading and support members of the test fixture that are in contact with the bearing cylinders.

8.3.2 Carefully place each test specimen into the test fixture to avoid possible damage and contamination and to ensure alignment of the test specimen relative to the test fixture. In particular, there should be an equal amount of overhang of the test specimen beyond the outer bearing cylinders, and the test specimen should be directly centered below the axis of the applied load. In some cases, depending on the fixture design, the test fixture/test specimen assembly is not securely in position but movable while being loaded into the load train of the test machine. In this case, a room-temperature adhesive may be used to hold the test specimen firmly in place relative to the bearing cylinders and the fixture members. However, care must be exercised to ensure that use of an adhesive shall not have any undesirable effect on slow crack growth (strength) of the test specimen through contamination and/or reaction by organic residue.

NOTE 18—Various room-temperature adhesives, such as an acetate household cement or a cyanoacrylate adhesive, may be utilized for this purpose if the adequacy of an adhesive (see 8.3.2), evaluated prior to testing, is met.

8.4 *Loading the Test Fixture/Specimen Assembly into Furnace*—Mount the test fixture/test specimen assembly in the load train of the test machine prior to heating the furnace. If necessary, use a preload of no more than 25 % of the fracture load to maintain system alignment. If uneven line loading of the test specimen occurs, use fully articulating fixtures.

NOTE 19—The temperature of the furnace during loading of the test fixture/test specimen assembly is not necessarily at room temperature. The furnace could be preheated or remain hot from the previous testing, with temperatures not affecting any undesirable thermal shock damage to test fixtures and test specimens. Appropriate precautions should be taken to ensure operator safety from the hazards of thermal or electrical burns. Safety gloves, safety glasses, or other safety tools, or combination therefore, are essential.

8.5 If test specimen deflection is to be measured (see 6.6) using a contact type of equipment, position the deflection-measurement probe(s) with its rounded tip in contact with the midpoint or the inner load points (tension side), or both, of the test specimen. Exercise care to apply an appropriate contact load (see Note 15).

8.6 Some appropriate means should be furnished for keeping test fragments from flying about the furnace after fracture. If possible, retrieve the test specimens from the furnace as soon as possible after fracture in order to preserve the primary fracture surfaces for subsequent fractographic analysis.

8.7 *Environment*—Choose the test environment as appropriate to the test program. If the test environment is other than ambient air or vacuum, supply the environmental chamber with the test medium so that the test specimen is completely exposed by the test environment. The consistent conditions (composition, supply rate, and so forth) of the test environment should be maintained throughout the tests (also refer to 6.5).

8.8 *Heating to the Test Temperature*—Heat the test specimen to the test temperature at the prescribed heating rate. Temperature overshoot over the test temperature shall be strictly controlled and shall be no more than 5°C. Maintain the temperature within± 5°C (soak time) to allow the entire system to reach thermal equilibrium. Prior to testing, the soak time should be determined experimentally at the test temperature.

8.9 *Hot-Furnace Loading and Heating (Optional)*—In some cases, test specimens may be loaded directly into a hot furnace, as described in 8.4 of Test Method C 1211. The fixture may be either left in the furnace for the entire time or removed partially or completely, depending on the details of the systems. Exercise care to ensure that the bearing cylinders and test specimen are positioned accurately. Furthermore, exercise *extreme care* to ensure that possible damage associated with thermal shock shall not have any effect on strength or slow crack growth, or both, of test specimens. If needed and possible, place the deflection-measurement probe in contact with the midpoint of specimens between the two inner bearing cylinders, in accordance with 8.5. Determine the soak time of the test specimen at the test temperature experimentally prior to testing.

8.10 *Preloading*:

8.10.1 The time required for any strength testing can be minimized by applying some preload to a test specimen prior to testing, provided that the strength determined with preloading does not differ from that determined without preloading. It has been shown that in constant stress-rate testing, considerable preloads can be applied to ceramic specimens with no change in the strength obtained, resulting in a significant reduction of test time **(15)**. The relationship between strength and preloading is as follows:

$$\sigma^* = (1 + \alpha_p^{n+1})^{\frac{1}{n+1}} \quad (1)$$

where:

σ^* = normalized strength = σ_{fp}/σ_{fn},
α_p = preloading factor ($0 \leq \alpha_p < 1.0$) = σ_o/σ_{fn},
σ_{fp} = strength with preloading,
σ_{fn} = strength without preloading,
σ_o = preload stress, and
n = slow crack growth parameter.

The strength with preloading is dependent both on the magnitude of preloading and on the SCG parameter n. The plots of the normalized strength as a function of preloading for different n's, Eq 1, are depicted in Fig. 3. This figure shows that, for example, a preload corresponding to 80 % ($= \alpha_p$) of strength for $n \geq 20$ (common to most ceramic materials at elevated temperatures) results in a maximum strength increase by 0.04 % ($\alpha_p = 1.0004$). And a preload of 70 % gives the maximum increase by 0.003 % ($\alpha_p = 1.00003$). This means that a considerable amount of test time can be saved through an appropriate choice of preloading (In this example, an 80 % saving of test time results from a preload of 80 %, and a 70 % saving from a preload of 70 %). It is suggested that strength (or fracture load) for a given test rate be first estimated using at least three specimens and then the preload be determined from Eq 1 or Fig. 3. For a conservative result, take the SCG parameter $n \geq 20$. The preload, of course, can be adjusted from specimen to specimen based on the converging strength data (to the mean) as well as the scatter of strength, as testing proceeds. Preloading can save the most test time when it is applied at the lowest test rate since most (> 80 %) of total test time is consumed at the lowest stress rate **(15)**. In summary, one may use Eq 1 or Fig. 3 as a guideline to apply an appropriate amount of preload to save test time, if desired. Preloading can be applied more accurately and quickly by using the load-controlled mode than the displacement-controlled mode.

8.10.2 Apply the predetermined preload to the test specimen within 20 s.

8.11 *Conducting the Test*—Initiate the data acquisition. Start the test mode.

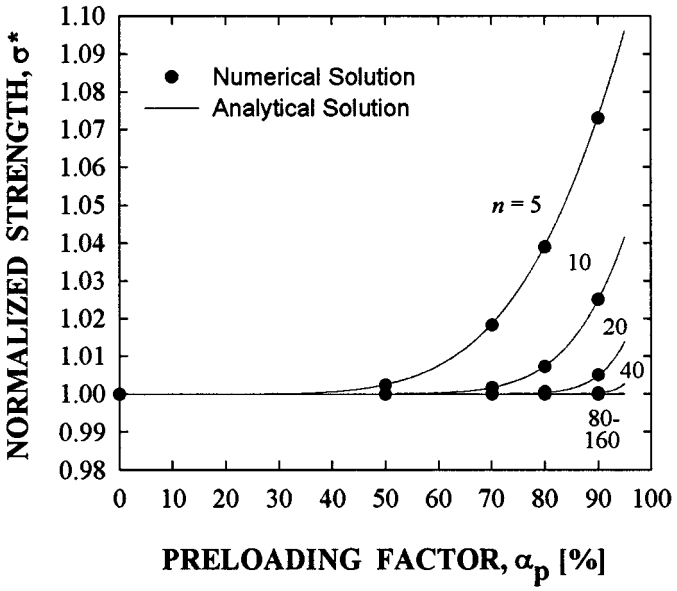


FIG. 3 Normalized Strength as a Function of Preloading for Different Slow Crack Growth Parameters *n*'s (15)

8.11.1 *Recording*—For either load-control or displacement-control mode, record a load versus time curve for each test in order to determine the actual loading rate, and thus to calculate the corresponding stress rate (see also 6.7 and 9.2). Determine the actual load rate in units of newtons per second from the slope of the load versus time curve for each test specimen. The initial nonlinear portion of the curve should not be used in determining the slope. The slope should be the tangent to the load-time data using an analog chart recorder when a high test rate is employed. Consider the curve including the portion at or near the point of fracture. Exercise care in recording adequate response-rate capacity of the recorder in this case, as described in 8.2 and Note 16. Also, record a deflection-time, or load-deflection curve, if determined, in accordance with 6.6.

8.11.2 *Nonlinearity in Load-Time (or Load-Deflection) Curve*—If nonlinearity is observed from the recorded load-time (or deflection-time or load-deflection) curves, creep deformation is probably present. Although it is difficult to specify a particular limit on creep deformation, it may be safe to limit a nominal maximum (tensile) creep strain to no more than 0.05 % **(10)**. Any other limit may be allowable, based on a mutual agreement, but this shall be stated in the report. Creep deformation may become dominant at lower test rate as well as at higher test temperature. If the creep strain is greater than an allowable limit, use a faster (typically one order of magnitude greater) test rate with the requirement of at least four different test rates still being met. In addition, apply some preload to the test specimen to shorten test time, thereby reducing overall creep deformation (see Note 7 and 8.10).

NOTE 20—In some cases, depending on material and test machine, no conditions may be found that meet the linearity and number of test rates criteria. In this case, the SCG parameters of the material may be evaluated only for reference information using the valid data points obtained. In an extreme case, this test method may not be applicable at all at certain higher temperatures because of significant creep deformation occurring in the entire range of test rates. If the limitations associated with creep deformation cannot be remedied in flexure testing, one, if desired, may utilize other testing such as "constant stress-rate tension testing" to characterize slow crack growth behavior of the material. However, note that no alternative elevated-temperature SCG testing, other than this test method, is currently available as a standard. It is generally recommended to follow the test procedures, test requirements, and guidance (except the specifications of test specimens and test fixtures) specified in this test method if other testing is to be performed.

8.11.2.1 *Creep Strain*—Use the following equations to estimate the corresponding nominal (not "true") creep strain from the results of various deflection-measurements (see 6.6).

Based on the midpoint deflection measurement:

$$\epsilon_{cr} = \frac{48d}{11L^2}\Delta y \tag{2}$$

Based on the inner load-point(s) measurement:

$$\epsilon_{cr} = \frac{6d}{L^2}\Delta y \tag{3}$$

Based on the deflection measurement of the midpoint relative to the two inner load points:

$$\epsilon_{cr} = \frac{16d}{L^2}\Delta y \tag{4}$$

where:

ϵ_{cr} = nominal maximum tensile creep strain of a flexure test specimen,
d = specimen depth, mm,
L = outer (support) span of the test fixture, mm, and
Δy = creep deflection, mm, corresponding to the nonlinear portion at failure, as depicted in Fig. 4.

NOTE 21—The previous equations, Eq 2-4, are based on the simple (elastic) beam theory, which also corresponds to the case when the stress exponent in creep is unity **(18)**.

8.11.3 *Fracture Load*—Measure fracture load with an accuracy of ±1.0 %.

8.11.4 Upon fracture, cool the test specimen and test apparatus to ambient temperature or to a predetermined temperature.

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

8.12 *Post-Test Treatments*:

8.12.1 Carefully collect all primary broken fragments. Clean with appropriate media if necessary and store in a protective container for further analysis such as fractography.

8.12.2 *Post-Test Specimen Dimensions*—Measure the thickness and width of each test specimen to within 0.002 mm, at a point near the fracture origin. In order to avoid damage to the test specimen prior to testing, it is generally recommended that measurements be made after fracture. In a special case where there is a concern about dimensional change of test specimens after testing due to reaction/reaction products, make the measurements prior to testing (refer to 7.3).

8.12.3 *Fracture Location*—Examine the location of fracture origin for each test specimen. Make certain that a valid test is one in which fracture occurs only in the uniformly stressed section (that is, the inner span).

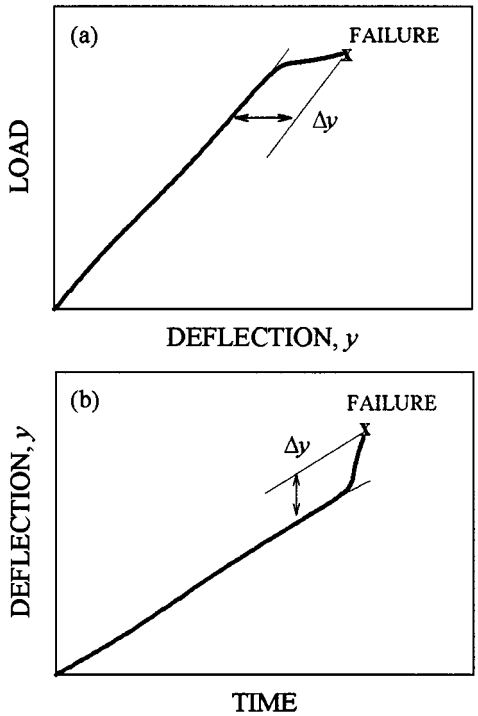


FIG. 4 Schematic Diagrams of Methods for Determining Creep Deflection: (a) Load-Deflection Curve; (B) Deflection-Time Curve

NOTE 22—Due to the nonuniform, steep stress-gradient occurring outside the inner span, it is rarely possible to determine the exact stress rate of a test specimen fractured outside the inner span. Therefore, the test specimens which fractured outside the inner span are not recommended for use as valid data points in determining the slow crack growth parameters. In the case of multiple fractures, it is recommended to ascertain that the primary fracture occurred inside the inner span. Guidance for determining primary fracture is given in Practice C 1322. From a conservative standpoint, when completing a required number of test specimens at each test rate, test one replacement test specimen for each test specimen that fractured outside the inner span. However, for more rigorous statistical analysis (such as Weibull statistics) with a large number of test specimens, a censoring technique can be used to deal with such anomalous data points as discussed in Practice D 1239.

8.12.4 *Fractography*—Fractographic analysis of fractured test specimens is highly recommended to characterize the types, locations, and sizes of fracture origins as well as the flaw extensions due to slow crack growth, if possible. Follow the guidance established in Practice C 1322.

9. Calculation

9.1 *Strength*:

9.1.1 The standard formula for the strength of a beam in four-point ¼-point flexure is as follows:

$$\sigma_f = \frac{3PL}{4bd^2} \tag{5}$$

where:

σ_f = flexural strength, MPa,
P = break load, N,
L = outer (support) span of the test fixture, mm,
b = test specimen width, mm, and
d = test specimen depth, mm.

9.1.2 Eq 5 shall be used for reporting the results and is the common equation used for the flexural strength of a test specimen. Thermal expansion effects on calculation are discussed in 9.4.

9.1.3 Based on individual strength data determined at each test rate (either applied nominal load rate for load-control mode or applied nominal displacement rate for displacement-control mode), calculate the corresponding mean strength, standard deviation, and coefficient of variation as follows:

$$\bar{\sigma}_f = \frac{\sum_{j=1}^{N} \sigma_j}{N} \tag{6}$$

$$SD_f = \sqrt{\frac{\sum_{j=1}^{N} (\sigma_j - \bar{\sigma}_f)^2}{N-1}} \tag{7}$$

$$CV_f(\%) = \frac{100(SD_f)}{\bar{\sigma}_f} \tag{8}$$

where:

$\bar{\sigma}_f$ = mean strength, MPa,
σ_j = jth measured strength value, MPa,
N = number of test specimens tested validly (that is, fractured in the inner span) at each test rate, a minimum of 10 test specimens,
SD_f = standard deviation, and

CV_f = coefficient of variation.

9.2 *Stress Rate*—The stress rate of each test specimen subjected to either displacement-control or load-control mode is calculated using the actual load rate determined (8.11.1) as follows:

$$\dot{\sigma} = \frac{3\dot{P}L}{4bd^2} \tag{9}$$

where:
$\dot{\sigma}$ = stress rate, MPa/s, and
$\dot{P}$ = load rate, N/s.

9.3 *Slow Crack Growth Parameters, n and D*:

9.3.1 A small variation of stress rate may occur from one test specimen to another even when subjected to the same test rate. Use each individual stress rate, not averaged per test rate, in determining SCG parameters. For each specimen tested, plot log (*flexural strength*) as a function of log (*stress rate*) (a flexural strength-stress rate diagram). The SCG parameters *n* and *D* can be determined by a linear regression analysis using all log σ_f (not averaged per test rate) over the complete range of individual log $\dot{\sigma}$ (not averaged per test rate), based on the following equation (see Appendix X1 for derivation):

$$\log \sigma_f = \frac{1}{n+1}\log \dot{\sigma} + \log D \tag{10}$$

Include in the log σ_f versus log $\dot{\sigma}$ diagram all the data points determined with valid tests. Examine the data points if a significant or obvious nonlinearity exists in the relationship between log (*flexural strength*) and log (*stress rate*) particularly at lower test (stress) rates, occurring for some materials presumably due to different failure mechanisms associated with enhanced creep or crack healing/blunting (see 5.4 and 5.5, Fig. 1). Estimate the strength value at the test rate where an obvious nonlinearity occurs, by extrapolating the regression line represented by the rest of the strength data. If a deviation of the "actual" mean strength value (exhibiting nonlinearity) from the "estimated" strength value (marked with "N" in Fig. 1b or 1c) by extrapolation is about or greater than 15 %, do not include such data in the regression analysis. The occurrence of significant strength degradation may also be identified by unique features such as the presence of micro- or macro-cracks, or both, in the tensile surface, and as excessive creep deformation of test specimens. A typical example of plot of log (*flexural strength*) as a function of (*stress rate*) (with no obvious nonlinearity) is shown in Fig. 5.

NOTE 23—This test method is intended to determine only slow crack growth parameters *n* and *D*. The calculation of the parameter, *A*, (in $v = A[K_I/K_{IC}]^n$) needs other material parameters, and is beyond the scope of this test method (see Appendix X1).

NOTE 24—The parameter *D* has units of $[(\text{MPa})^n\text{s}]^{1/(n+1)}$ with stress rate in MPa/s and strength in MPa, while the parameter *n* is nondimensional.

NOTE 25—This test method is primarily for test specimens with inherent natural flaws. If test specimens, however, possess any residual stresses (which would not have been annealed out at test temperatures) produced by localized contact damage (for example, particle impact or indents) or any other treatments, the estimated SCG parameters should be differentiated by denoting them as n' and D' instead of *n* and *D*. Refer to Ref **(8)** for more detailed information on the analysis of slow crack growth behavior of a material containing a localized residual stress field.

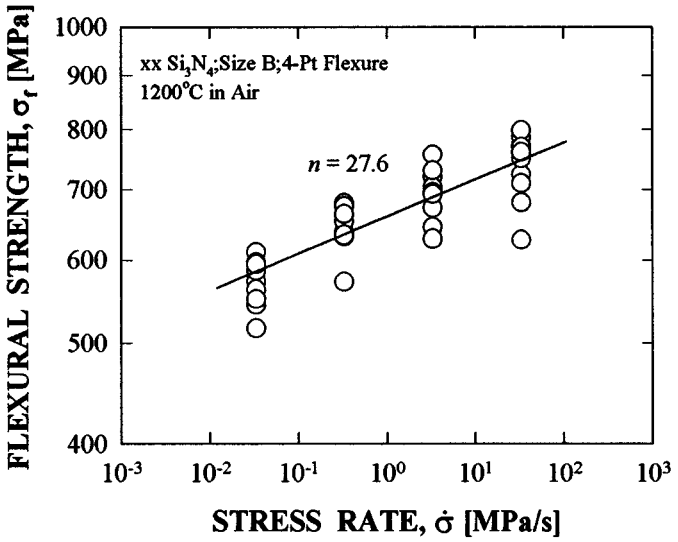


FIG. 5 Schematic of a Flexural Strength-Stress Rate Diagram, a Plot of Log (*Flexural Strength*) Versus Log (*Stress Rate*)

9.3.1.1 Calculate the slope of the linear regression line as follows:

$$\alpha = \frac{K\sum_{j=1}^{K}(\log \dot{\sigma}_j \log \sigma_j) - (\sum_{j=1}^{K}\log \dot{\sigma}_j)(\sum_{j=1}^{K}\log \sigma_j)}{K\sum_{j=1}^{K}(\log \dot{\sigma}_j)^2 - (\sum_{j=1}^{K}\log \dot{\sigma}_j)^2} \tag{11}$$

where:
α = slope,
$\dot{\sigma}_j$ = *j*th measured stress rate, MPa/s,
σ_j = *j*th measured strength value, MPa, and
K = total number of test specimens tested validly for the whole series of tests, a minimum of 40 specimens with four test rates.

9.3.1.2 Calculate the SCG parameter *n* as follows:

$$n = \frac{1}{\alpha} - 1 \tag{12}$$

9.3.1.3 Calculate the intercept of the linear regression line as follows:

$$\beta = \frac{(\sum_{j=1}^{K}\log\sigma_j)\sum_{j=1}^{K}(\log\dot{\sigma}_j)^2 - (\sum_{j=1}^{K}\log\dot{\sigma}_j \log\sigma_j)(\sum_{j=1}^{K}\log\dot{\sigma}_j)}{K\sum_{j=1}^{K}(\log\dot{\sigma}_j)^2 - (\sum_{j=1}^{K}\log\dot{\sigma}_j)^2} \tag{13}$$

where:
β = intercept.

9.3.1.4 Calculate the SCG parameter *D* as follows:

$$D = 10^{\beta} \tag{14}$$

9.3.1.5 Calculate the standard deviations of the slope α and of the SCG parameter *n* as follows:

$$SD_\alpha = \sqrt{\frac{K}{K-2}\frac{\sum_{j=1}^{K}(\alpha \log \dot{\sigma}_j + \beta - \log \sigma_j)^2}{K\sum_{j=1}^{K}(\log \dot{\sigma}_j)^2 - (\sum_{j=1}^{K}\log \dot{\sigma}_j)^2}} \tag{15}$$

$$SD_n = \frac{SD_\alpha}{\alpha^2} \tag{16}$$

where:
SD_α = standard deviation of the slope α, and
SD_n = standard deviation of the SCG parameter *n*.

9.3.1.6 Calculate the standard deviations of the intercept β and of the SCG parameter *D* as follows:

$$SD_\beta = \sqrt{\frac{\sum_{j=1}^{K}(\alpha \log \dot{\sigma}_j + \beta - \log \sigma_j)^2 \sum_{j=1}^{K}(\log \dot{\sigma}_j)^2}{(K-2)[K\sum_{j=1}^{K}(\log \dot{\sigma}_j)^2 - (\sum_{j=1}^{K}\log \dot{\sigma}_j)^2]}} \quad (17)$$

$$SD_D = 2.3026\,(SD_\beta)(10^\beta) \quad (18)$$

where:
SD_β = standard deviation of the intercept β, and
SD_D = standard deviation of the SCG parameter *D*.

9.3.1.7 Calculate the coefficients of variation of the SCG parameter *n* and of the SCG parameter *D* as follows:

$$CV_n\,(\%) = \frac{100(SD_n)}{n} \quad (19)$$

$$CV_D\,(\%) = \frac{100\,(SD_D)}{D} \quad (20)$$

where:
CV_n = coefficient of variation of the SCG parameter *n*, and
CV_D = coefficient of variation of the SCG parameter *D*.

9.3.1.8 Calculate the square of correlation coefficient (*r*) of the linear regression line as follows:

$$r^2 = \frac{[K\sum_{j=1}^{K}(\log \dot{\sigma}_j \log \sigma_j) - (\sum_{j=1}^{K}\log \dot{\sigma}_j \sum_{j=1}^{K}\log \sigma_j)]^2}{[K\sum_{j=1}^{K}(\log \dot{\sigma}_j)^2 - (\sum_{j=1}^{K}\log \dot{\sigma}_j)^2][K\sum_{j=1}^{K}(\log \sigma_f)^2 - (\sum_{j=1}^{K}\log \sigma_f)^2]} \quad (21)$$

where:
r^2 = square of the correlation coefficient.

NOTE 26—The sources and basis of the preceding equations (Eq 15-21) are from Refs **(19, 20)**. The standard deviations in Eq 15-18 are also called standard error in the literature.

NOTE 27—For a better representation of SCG behavior of the material, it is recommended that the estimated regression line with the determined value of SCG parameter n be included in the flexural strength-stress rate diagram, not extended beyond the data by more than ½ decade of stress rate at either end of the data, as shown in Fig. 5. In addition, some key information such as test material, test temperature, test specimen size, test fixture, and test environment, and so forth, may be included in the diagram for the sake of completeness (see Fig. 5).

9.4 *Alternate Practice*—Eq 5 and Eq 9 do not account for the effects of thermal expansion of the test fixture and test specimen since all dimensions are taken at room temperature. Thermal expansion of the test fixture and test specimen can lead to flexure stress and stress rate errors of 1 to 3 % for advanced ceramics. The use of the corrected equations will not affect the SCG parameter *n*; whereas, it will affect the SCG parameter *D*. Refer to Annex A.1 of Test Method C 1211 for the equation accounting for such thermal expansion. The use of the thermal-expansion corrected equation must be stated explicitly in the report.

10. Report

10.1 *Test Specimens, Equipments, and Test Conditions*—Report the following information for the test specimens, equipment, and test conditions. Note in the report any deviations and alterations from the procedures and requirements described in this test method.

10.1.1 Date and location of tests.

10.1.2 Specimen geometry type and specimen dimensions.

10.1.3 All relevant material data including vintage data or billet identification data. As a minimum, the date the material was manufactured must be reported.

10.1.4 Exact method of test specimen preparation, including all stages of machining.

10.1.5 Heat treatments or heat exposures, if any.

10.1.6 Relevant information on randomization of the test specimens.

10.1.7 Methods of test specimen cleaning and storage.

10.1.8 All preconditioning of test specimens prior to testing, if any.

10.1.9 Type of configuration of the test machine including the load cell.

10.1.10 Type, configuration and material of the test fixture with degree of articulation.

10.1.11 Type and configuration of the data acquisition system.

10.1.12 Test temperature and test environment (type, conditions, and application method).

10.1.13 Method of loading the test specimens into the furnace, and method of the test specimen/test fixture assembling.

10.1.14 Rate of heating, soak time, and cooling rate of the furnace (if any).

10.1.15 Ambient conditions such as temperature and humidity.

10.1.16 Method and magnitude of preloading for each test specimen, if any.

10.1.17 Test mode (load or displacement control), number of test rates, and test rates.

10.2 *Test Results*—Report the following information for the test results. Note in the report any deviations and alterations from the procedures and requirements described in this test method.

10.2.1 Number of the valid tests (for example, fracture in the inner span) as well as of the invalid tests (for example, fracture outside the inner span) at each test rate.

10.2.2 Equations used for stress and stress rate, and in particular, whether the thermal expansion of the test fixture and test specimen was taken into account.

10.2.3 Actual load rate and stress rate of each test specimen to three significant figures.

10.2.4 Strength of each test specimen to three significant figures.

10.2.5 Mean strength, standard deviation, and coefficient of variation determined at each test rate (9.1.3).

10.2.6 Graphical representation (Fig. 5) of test results showing log (*flexural strength*) as a function of log (*stress rate*) using all data points to meet the test requirements. Include in the figure the determined best-fit, linear regression line together with the estimated value of SCG parameter *n*. Include, if desired, in the figure some key information on test material,

test temperature, test specimen size, test fixture, and test environment, and so forth as shown in Fig. 5.

10.2.7 Slow crack growth parameters *n* and *D*, their standard deviations (*SD*'s) and coefficients of variation (*CV*'s), and the square (r^2) of the correlation coefficient of the linear regression line.

10.2.8 Allowable creep strain, and amount of nonlinearity (that is, creep strain) determined from test specimens and Eq 12-14, based on the load-deflection or deflection-time curve, if determined.

10.2.9 Any pertinent fractography information including type, location, and size of fracture origin as well as the degree of slow crack growth, if possible.

10.2.10 Any pertinent information on nonlinearity occurring in the relationship between log (*flexural strength*) and log (*stress rate*), if any.

11. Precision and Bias

11.1 The flexural strength of an advanced ceramic for a given test rate is not a deterministic quantity, but will vary from test specimen to test specimen. There will be an inherent statistical scatter in the results for finite sample sizes (for example, 30 specimens). Weibull statistics can model this variability as discussed in Practice C 1239. This test method has been devised so that the precision is high and the bias is low compared to the inherent variability of strength of the material.

11.2 The experimental stress errors as well as the error due to cross-section reduction associated with chamfering the edges have been analyzed in detail in Ref **(5)** and described in terms of precision and bias in 11.2 of Test Method C 1211.

11.3 The effects of thermal expansion have not been incorporated into Eq 2 and Eq 6 for flexure stress and stress rate. This typically will lead to an error in flexure strength and stress rate on the order of 1 to 3 %. (Only the parameter *D* will be affected; whereas, the parameter *n* will be unaffected.) If an adjustment due to thermal expansion is made, report this explicitly (see 9.4). Also refer to 11.2.5 and Annex A.1 of Test Method C 1211 for detailed information regarding calculation.

11.4 The statistical reproducibility of slow crack growth parameters determined from the constant stress-rate testing has been analyzed in detail **(2)**. The degree of reproducibility of SCG parameters depends on not only the number of test specimens but other experimental test variables. These variables include SCG parameters (*n* and *D*), Weibull modulus, and the number and range of test rates. For example, using the number and range of test rates recommended in this test method, for an advanced ceramic with a Weibull modulus of 12, a coefficient of variation of 10 % in *n* requires about 50 and 200 specimens in total, respectively, for $n = 20$ and 40. For a coefficient of variation of 20 % in *n*, the number of specimens can be reduced to about 20 and 60, respectively.

11.5 Bias may result from inadequate use and/or treatments of the test environment, particularly in terms of its composition, aging, and contamination.

12. Keywords

12.1 advanced ceramics; constant stress-rate testing; elevated temperatures; flexural strength; flexural testing; four-point flexure; slow crack growth; slow crack growth parameters

APPENDIX

(Nonmandatory Information)

X1. DERIVATION OF STRENGTH AS A FUNCTION OF APPLIED STRESS RATE IN CONSTANT STRESS-RATE TESTING ("DYNAMIC FATIGUE" EQUATION) (REFS (1, 21))

X1.1 For most ceramics and glasses, slow crack growth rate can be approximated by the empirical power-law relation **(21,22)**:

$$v = \frac{da}{dt} = A\left[\frac{K_I}{K_{IC}}\right]^n \tag{X1.1}$$

where:

v = slow crack growth rate,
a = crack length,
t = time,
A and n = slow crack growth parameters,
K_I = mode I stress intensity factor, and
K_{IC} = fracture toughness under Mode I condition.

X1.2 For a uniform remote applied stress (Mode I), the stress intensity factor can be expressed as follows:

$$K_I = Y\sigma\sqrt{a} \tag{X1.2}$$

where:

Y = geometry factor related to flaw shape and its orientation with respect to the direction of applied stress.

Using Eq X1.1-X1.2 with some manipulations, a relationship between the inert strength (σ_i) and the fracture strength (σ_f) under slow crack growth can be determined as follows:

$$\sigma_f^{n-2} = \sigma_i^{n-2} - \frac{1}{B}\int_0^t [\sigma(t)]^n dt \tag{X1.3}$$

where:

$$B = \frac{2K_{IC}^2}{AY^2(n-2)} \tag{X1.4}$$

= material/environment parameter.

X1.3 For constant stress-rate testing, $\sigma(t) = \dot{\sigma}\, t$, Eq X1.3 becomes:

$$\sigma_f^{n+1} = B(n+1)\,\sigma_i^{n-2}\,\dot{\sigma} \quad \text{(X1.5)}$$

In deriving Eq X1.5, it was assumed that $(\sigma_f/\sigma_i)^{n-2} << 1$ since $n \geq 5$ for most advanced ceramics. Now taking logarithm for both sides of Eq X1.5 yields:

$$\log \sigma_f = \frac{1}{n+1}\log \dot{\sigma} + \log D \quad \text{(X1.6)}$$

where:

$$\log D = \frac{1}{n+1}\log[B(n+1)\sigma_i^{n-2}] \quad \text{(X1.7)}$$

Therefore, the slow crack growth parameters n and D can be determined by a linear regression analysis based on Eq X1.6 when log (*flexural strength*) is plotted as a function of log (*stress rate*).

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