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Designation: C 1368 – 00

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

This standard is issued under the fixed designation C 1368; 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 ambient temperature. 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 Definitions E 1150, 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 (See Definitions E 1150).

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

1.3 *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

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

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

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

E 337 Test Method for Measuring Humidity with a Psychrometer (the Measurement of Wet-Bulb and Dry-Bulb Temperatures)[5]

E 380 Practice for Use of the International System of Units (SI) (The Modernized Metric System)[6]

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

2.2 *Military Handbook:*

MIL-HDBK-790 Fractography and Characterization of Fracture Origins in Advanced Structural Ceramics[7]

3. Terminology

3.1 *Definitions*—The terms described in Terminology C 1145, Terminology E 6, Terminology E 616, and Definitions E 1150 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)**

3.1.2 *constant stress rate,* $\dot{\sigma}$, *n*—a constant rate of maximum stress applied to a specified beam by using either a constant loading 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*—the container of bulk volume surrounding a test specimen. **(E 1150)**

3.1.5 *flexural strength,* σ_f, *n*—a measure of the 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 curve, n*—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: $\log \sigma_f = 1/(n + 1) \log \dot{\sigma} + \log D$. (See Appendix X1.)

[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 11.03.

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

[7] Available from Army Research Laboratory—Materials Directorate, Aberdeen Proving Ground, MD 21005.

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NOTE 3—In the ceramics literature, this is often called a dynamic fatigue curve.

3.1.7 *flexural strength-stress rate diagram, n*—a plot of flexural strength against stress rate. Both flexural strength and stress rate are plotted on log-log scales.

3.1.8 *fracture toughness, n*—a generic term for measures of resistance to extension of a crack. **(E 616)**

3.1.9 *inert flexural strength, 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.

NOTE 4—An inert condition may be obtained by using vacuum, low temperatures, very fast test rates, or any inert mediums.

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, n*—the magnitude of the ideal-crack-tip stress field (stress-field singularity) subjected to mode I loading in a homogeneous, linear elastic body. **(E 616)**

3.2 *Definition of Term Specific to This Standard:*

3.2.1 *slow crack growth parameters, n and D, n*—the parameters estimated as constants in the flexural strength-stress rate equation, which represent the degree of slow crack growth susceptibility of a material. (See Appendix Appendix X1.)

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 SCG. This test method provides the empirical parameters for appraising the relative SCG susceptibility of ceramic materials under specified environments. Furthermore, this test method may establish the influences of processing variables and composition on SCG 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.

4.2 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 of the beam thickness.

4.3 The specimen sizes and fixtures were chosen in accordance with Test Method C 1161, which provides a balance between practical configurations and resulting errors, as discussed in Refs **(4, 5)**. Only the four-point test configuration is used in this test method.

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

NOTE 5—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 (Ref **(6)**). It is generally accepted that actual data cannot reliably distinguish between the various formulations. Therefore, the mathematical analysis in this test method does not cover such alternative crack velocity formulations.

4.5 The mathematical relationship between flexural strength and stress rate was derived based on the assumption that the slow crack growth parameter is at least $n \geq 5$ (Refs **(1, 7, 8)**). Therefore, if a material exhibits a very high susceptibility to SCG, that is, $n < 5$, special care should be taken when interpreting the results.

4.6 The mathematical analysis of test results in accordance with the method in 4.4 assumes that the material displays no rising *R*-curve behavior. It should be noted that the existence of such behavior cannot be determined from this test method.

4.7 Slow crack growth behavior of ceramic materials exposed to stress-corrosive gases or liquid environments can vary as a function of mechanical, material, and electrochemical 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, SCG that is determined from the flexural strengths of a ceramic material is also a probabilistic phenomenon. Hence, a proper range and number of applied stress rates in conjunction with an appropriate number of specimens at each applied stress rate are required for statistical reproducibility and design (Ref **(2)**). Guidelines are provided in this test method.

NOTE 6—For a given ceramic material/environment system, the SCG parameter n is constant regardless of specimen size although its reproducibility is dependent on the variables mentioned in 4.8. By contrast, the SCG parameter D depends significantly on strength and thus on specimen size (see Eq X1.6 in Appendix X1).

4.9 The strength of a ceramic material for a given specimen and test fixture configuration is dependent on its inherent resistance to fracture, the presence of flaws, 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 or MIL-HDBK-790, or both).

5. Interferences

5.1 SCG may be the product of both mechanical and chemical driving forces. The chemical driving force for a given material with given flaw configurations can strongly vary with the composition, pH, and temperature of a test environment. Note that SCG testing is very time-consuming: it may take several weeks to complete testing 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 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 (>2000 MPa/s) may remain unchanged so that a plateau is observed in the plot of strength versus stress rate (Ref **(7)**). 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 excluded as data points in estimating the SCG parameters of the material. This test method addresses for this factor by recommending that the highest stress rate by ≤2000 MPa/s.

NOTE 7—The strength plateau of a material can be checked by measuring an inert flexural strength in an appropriate inert medium.

5.3 Surface preparation of test specimens can introduce fabrication flaws which may have pronounced effects on SCG behavior. 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 slow crack growth as well as in strength behavior.

6. Apparatus

6.1 *Testing Machine*—Testing machines used for this test method shall conform to the requirements of Practices E 4. Specimens may be loaded in any suitable testing machine provided that uniform test rates, either using load-controlled or displacement-controlled mode, can be maintained. The loads used in determining flexural strength shall be accurate within ±1.0 % at any load within the selected load rate and load range of the testing machine as defined in Practices E 4. The testing 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-controlled mode and from 10^{-7} to 10^{-4} m/s for displacement-controlled mode.

6.2 *Test Fixtures*—The configurations and mechanical properties of test fixtures should be in accordance with Test Method C 1161. 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 react with or contaminate the environment.

NOTE 8—For testing in water, for example, it is recommended that the test fixture be fabricated from stainless steel which is effectively inert to water. The bearing cylinders may be machined from hardenable stainless steel (for example, 440C grade) or a ceramic material such as silicon nitride, silicon carbide, or alumina.

6.2.1 *Four-Point Flexure*—The four-point-¼ point fixture configuration as described in 6.2 of Test Method C 1161 shall be used in this test method.

6.2.2 *Bearing Cylinders*—The requirements of dimensions and mechanical properties of bearing cylinders as described in 6.4 of Test Method C 1161 shall be used in this test method. It should be noted that the bearing cylinders shall be free to rotate in order to relieve frictional constraints, as described in 6.4.4 of Test Method C 1161.

6.2.3 *Semiarticulating Four-Point Fixture*—The semiarticulating four-point fixture as described in 6.5 of Test Method C 1161 may be used in this test method. This fixture shall be used when the parallelism requirements of test specimens are met in accordance with 7.1 of Test Method C 1161.

6.2.4 *Fully Articulating Four-Point Fixture*—The fully articulating four-point fixture as described in 6.6 of Test Method C 1161 may be used in this test method. Specimens which do not meet the parallelism requirements of 7.1 of Test Method C 1161, due to the nature of fabrication process (as-fired, heat-treated, or oxidized), shall be tested in this fully articulating fixture.

6.2.5 *Compliance of Test Fixture*—The test fixtures shall be stiffer than the specimen, so that most of the crosshead or actuator travel is imposed onto the specimen.

6.3 *Data Acquisition*—Accurate determination of both fracture load and test time is important since it affects 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. Ideally, 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 should be accurate to 1.0 % of the recording range and should have a minimum data acquisition rate of 1000 Hz (or 1 KHz) with a response of 5000 Hz (or 5 KHz) deemed more than sufficient. The appropriate data acquisition rate depends on the test rate; the higher the test rate the higher the acquisition rate, and vise versa.

6.4 *Environmental Facility*—If testing is conducted in any 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. The chamber should be large enough to fully immerse the test specimens in the environment, particularly for liquid environments. A circulation system to replenishment the test environment may be desirable. It should provide continuous filtration of the test medium in order to remove foreign debris and corrosive product. Additionally, the facility shall be able to safely contain the test environment.

7. Test Specimen

7.1 *Specimen Size*—The types and dimensions of rectangular beam specimens as described in 7.1 of Test Method C 1161 shall be used in this test method.

7.2 *Specimen Preparation*—Specimen fabrication and preparation methods as described in 7.2 of Test Method C 1161 shall be used in this test method.

7.3 *Handling, Cleaning, and Storage*—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. Clean test specimens with an appropriate cleaning medium such as methanol or high-purity (>99 %) isopropyl alcohol, since surface contamination of test specimens by lubricant, residues, rust, or dirt might affect slow crack growth behavior for certain test environments. After cleaning and drying, store test specimens in vacuum or desiccators to minimize or to avoid exposure to moisture in air. This is particularly important if testing is carried out in any environment other than ambient air or water. Moisture entrapped in specimen surfaces may result in accelerated SCG.

7.4 *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 applied stress rates, range of applied stress 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 stress rate is recommended in this test method. The total number of test specimens shall be at least 40, with at least four applied stress rates. The number of specimens (and stress rates) recommended in this test method has been established with the intent of determining not only reasonable confidence limits on both strength distribution and SCG parameters but also to help discern multiple-flaw populations.

NOTE 9—Refer to Ref **(2)** when a specific purpose is sought for the statistical reproducibility of SCG parameters.

8. Procedure

8.1 Choose the appropriate fixtures for the specific testing configurations (see Section 6 of Test Method C 1161). Use the four-point A fixture for the size A specimens. Similarly, use the B fixture for B specimens and the C fixture for 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 testing machine. Since various types of testing 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. The upper limits of the test rates of testing machines are controlled by 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 testing 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^{-4} m/s, respectively, for load- and displacement-controlled modes.

8.2.2 For a testing machine equipped with load-controlled mode, choose at least four loading 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 the testing machine equipped with displacement-controlled 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). However, for better statistical reproducibility of SCG parameters, the use of five or more test rates (evenly spaced in a logarithmic scale) covering four or more orders of magnitude is recommended if the testing machine is capable and the specimens are available. In general, the load-controlled mode yields a better output wave-form than the displacement-controlled mode, particularly at low test rates. In addition, the specified applied loading rate can be directly related with stress rate, regardless of the system compliance of test frame, load train, fixture and specimen, thus simplifying data analysis. In the displacement-controlled mode, however, the loading rate to be determined is a function of both applied displacement rate and system compliance so that the actual loading rate should always be measured and used to calculate a corresponding stress rate, thus making data analysis complex. Therefore, a load-controlled test is the preferred test mode.

NOTE 10—When using the faster test rates, care must be exercised particularly for the conventional, older electromechanical testing machines equipped with slow-response load cells and chart recorders. Such machines have 100 MPa/s as an upper limit stress rate at which the chart recorder or the load cell, or both, cannot follow load increase and hence cannot correctly monitor the fracture load (Refs **(9, 10)**). 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 ($\geq$3 s). However, the use of a better load cell (or piezoelectric load cell) or a fast-response chart recorder, or both, or a digital data acquisition system can improve the existing performance so that higher test rates (up to 2000 MPa/s Ref **(9)** can be achieved. It has been shown that the digitally-controlled, modern testing machine is capable of applying stress rates up to 10^{5} MPa/s (Ref **(8)**).

8.3 Carefully place each specimen into the test fixture to preclude possible damage and contamination and to ensure alignment of the specimen relative to the test fixture. In particular, there should be an equal amount of overhang of the specimen beyond the outer bearing cylinders and the specimen should be directly centered below the axis of the applied load. Assemble the test fixture/specimen in the testing machine. Mark the specimen to identify the points of load application and also so that the tensile and compression faces can be distinguished. Carefully drawn pencil marks will suffice.

8.4 Slowly apply an initial preload of not more than 20 N to the specimen by means of the fixture. Inspect the points of contact between the bearing cylinders and the specimen to ensure even line loading. If uneven line loading of the specimen occurs, use fully articulating fixtures.

8.5 *Environment*—Choose the test environment as appropriate to the test program. Fill the clean environmental chamber with the test medium so that the specimen is completely immersed in or surrounded by the test environment. The

immersion or exposure time for equilibration of the test specimen in the environment should be determined by agreement between the parties involved in the test program. This is particularly important for environments which are chemically corrosive to the specimen. The environment should be consistent for the test series and should be reported. If the tests are carried out in a humid atmosphere, the relative humidity shall not vary by more than 10 % during the entire test series. Determine the relative humidity in accordance with Test Method E 337.

NOTE 11—If it is necessary to precondition the test specimens in an environment prior to testing, such as aging in water, the preconditioning parameters (temperature, time, solution, and so forth) should be consistent for all the test specimens and should be reported.

8.6 *Preloading:*

8.6.1 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, considerably high preloading can be applied to ceramics specimens with no change in the strength obtained, resulting in a significant reduction of test time (Refs **(11, 12)**). 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. 1. This figure shows that, for example, a preload corresponding to 80 % ($= \alpha_p$) of strength for $n \geq 20$ (common to most glass and ceramic materials in water) results in a maximum strength increase by 0.04 % ($\sigma^* \leq 1.00004$). And a preload of 70 % gives the maximum increase by 0.003 % ($\sigma^* \leq 1.00003$). This means that a considerable amount of test time can be saved through an appropriate choice of preloading (in this example, a 80 % saving of test time results from a preload of 80 %, and a 70 % saving from a preload of 70 %). It is suggested that an approximate 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. 1. 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 stress rate since most (>80 %) of total test time is consumed at the lowest stress rate (Refs **(11, 12)**). In summary, one may use Eq 1 or Fig. 1 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 rather than the displacement-controlled mode.

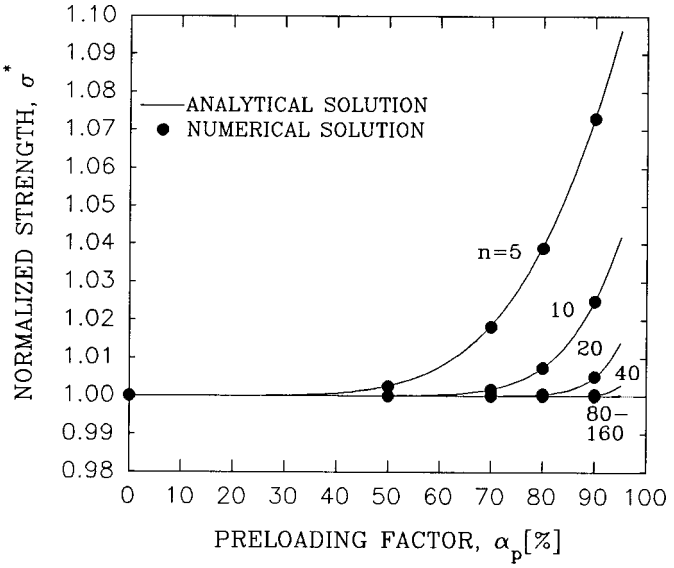


FIG. 1 Normalized Strength as a Function of Preloading for Different Slow Crack Growth Parameter *n*'s (Ref (11))

8.6.2 Apply the predetermined preload to the specimen within a few seconds.

8.7 For either load-controlled 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 6.3 and 9.2). The actual loading rate in units of newtons per second should be determined from the slope of the load versus time curve for each specimen. The slope should be the tangent to the curve including the portion at or near the point of fracture. Care should be taken in recording the load-time data using an analog chart recorder when a high test rate is employed. Consider the adequate response-rate capacity of the recorder in this case, as described in 8.2 and Note 10.

8.8 When tests are conducted in ambient air, put cotton, crumbled tissues, or other appropriate material around the specimen to prevent pieces from flying out of the fixtures upon fracture. When a corrosive liquid environment is used, put a proper protective cover onto the environmental chamber to keep the test environment from splashing out of the chamber upon specimen fracture.

8.9 *Breakload*—Measure fracture load with an accuracy of ±1.0 %.

8.10 *Post-Test Treatments:*

8.10.1 Collect all primary broken fragments. Thoroughly clean with an appropriate medium and completely dry them in an oven or a vacuum chamber, particularly when the specimen has been tested in a corrosive environment. It is highly recommended to retain and preserve all the primary fracture fragments for further analysis such as fractography.

8.10.2 *Specimen Dimensions*—Measure the thickness and width of each test specimen to within 0.0025 mm. In order to avoid damage to the specimen, it is recommended that measurement be made after fracture at a point near the fracture origin.

8.10.3 Measure and report the fracture location relative to the midpoint of the gage length (the uniform stressed section, that is, the inner span). The convention used should be that the midpoint of the gage length is 0 mm with positive (+) measurements toward the right of the specimen as tested (and marked) and negative (–) measurements toward the left of the specimen as tested (and marked).

8.10.4 Note that the specimens broken outside the gage length are not recommended for use as valid data points in determining the SCG parameters. Results from the specimens broken outside the gage length are considered not only anomalous but ambiguous or uncertain particularly in the determination of exact, corresponding stress rates of those specimens. This is mainly due to the nonuniform, steep stress-gradient occurring outside the gage length. From a conservative standpoint, when completing a required number of specimens at each test rate, test one more replacement specimen for each specimen that is broken outside the gage length. 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 C 1239.

8.10.5 *Fractography*—Fractographic analysis of failed 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 guidelines established in Practice C 1322 or MIL-HDBK-790, or both.

8.11 Clean the test fixtures, if necessary, and repeat the test on a new test specimen. Check the condition/adequacy of the test environment for further use.

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:

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

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

9.1.2 Eq 2 shall be used for reporting the results and is the common equation used for the flexural strength of a specimen.

NOTE 12—It should be recognized, however, that Eq 2 does not necessarily give the stress that was acting directly upon the flaw associated with failure. In some instances, the fracture stress as well as the stress rate must be corrected for subsurface origins and flaw extensions.

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

$$\bar{\sigma}_f = \frac{\sum_{j=1}^{N} \sigma_j}{N} \tag{3}$$

$$SD_f = \sqrt{\frac{\sum_{j=1}^{N} (\sigma_j - \bar{\sigma}_f)^2}{N-1}} \tag{4}$$

$$CV_f\,(\%) = \frac{100\,(SD_f)}{\bar{\sigma}_f} \tag{5}$$

where:
$\bar{\sigma}_f$ = mean strength, MPa,
σ = measured value, MPa,
N = number of specimens tested validly (that is, fracture in the gage length) at each test rate, a minimum of 10 specimens,
SD_f = standard deviation, and
CV_f = coefficient of variation.

9.2 *Stress Rate:*

9.2.1 The stress rate of each specimen subjected to either displacement-controlled or load-controlled mode is calculated using the actual loading rate determined (8.7) as follows:

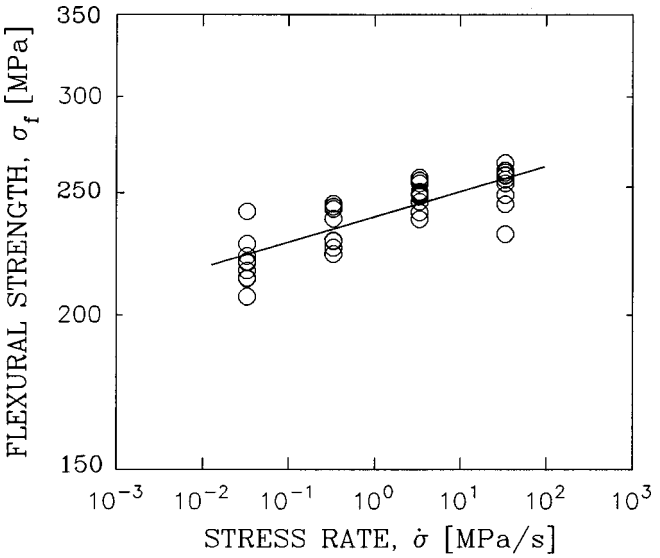
$$\dot{\sigma} = \frac{3\dot{P}L}{4bd^2} \tag{6}$$

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

9.2.2 A small variation of stress rate may occur from one specimen to another even when subjected to the same test rate. Use each individual stress rate (not averaged per test rate) in determining the SCG parameters.

9.3 *SCG Parameters, n and D:*

9.3.1 For each stress rate, plot log σ_f versus log $\dot{\sigma}$ (a flexural strength-stress rate diagram), as shown in Fig. 2. The SCG



NOTE 1—The best-fit regression line, a flexural strength-stress rate curve, determined based on the linear regression analysis using all the data points is included.

FIG. 2 Schematic of a Flexural Strength-Stress Rate Diagram, a Plot of Log (Flexural Strength) Versus Log (Stress Rate)

parameters n and D can be determined by a linear regression analysis (Ref **(13)**) using all log strength values (not averaged per test rate) over the complete range of individual log stress rates (not averaged per test rate), based on the following equation (see Appendix X1 for derivation):

$$log\ \sigma_f = \frac{1}{n+1} log\ \dot{\sigma} + log\ D \tag{7}$$

NOTE 13—This test method is intended to determine only SCG parameters n and D. The calculation of the parameter A needs other material parameters and is beyond the scope of this test method (see Appendix X1).

NOTE 14—This test method is primarily for specimens with inherent natural flaws. If the test specimens, however, possess any residual stresses 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'. Refer to Ref **(7)** for more detailed information on the analysis of slow crack growth behavior of a material containing a residual stress field.

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

$$\alpha = \frac{K\sum_{j=1}^{K}(log\ \dot{\sigma}_j\ log\ \sigma_j) - (\sum_{j=1}^{K} log\ \dot{\sigma}_j \sum_{j=1}^{K} log\ \sigma_j)}{K\sum_{j=1}^{K}(log\ \dot{\sigma}_j)^2 - (\sum_{j=1}^{K} log\ \dot{\sigma}_j)^2} \tag{8}$$

where:
α = slope, and
K = total number of 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:

$$n = \frac{1}{\alpha} - 1 \tag{9}$$

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

$$\beta = \frac{(\sum_{j=1}^{K} \log\sigma_j)\sum_{j=1}^{K}(\log\dot{\sigma}_j)^2 - (\sum_{j=1}^{K}\log\dot{\sigma}_j \log\sigma_j)(\sum_{j=1}^{K} \log\dot{\sigma}_j)}{K\sum_{j=1}^{K}(\log\dot{\sigma}_j)^2 - (\sum_{j=1}^{K} \log\dot{\sigma}_j)^2} \tag{10}$$

where:
β = intercept.

9.3.1.4 Calculate the SCG parameter D as follows:

$$D = 10^{\beta} \tag{11}$$

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

$$SD_\alpha = \sqrt{\frac{K}{K-2}\frac{\sum_{j=1}^{K}(\alpha \log\ \dot{\sigma}_j + \beta - \log\ \sigma_j)^2}{K\sum_{j=1}^{K}(\log\ \dot{\sigma}_j)^2 - (\sum_{j=1}^{K} \log\ \dot{\sigma}_j)^2}} \tag{12}$$

$$SD_n = \frac{SD_\alpha}{\alpha^2} \tag{13}$$

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:

$$SD_\beta = \sqrt{\frac{\sum_{j=1}^{K}(\alpha \log\ \dot{\sigma}_j + \beta - \log\ \sigma_j)^2 \sum_{j=1}^{K}(\log\ \dot{\sigma}_j)^2}{(K-2)\,[K\sum_{j=1}^{K}(\log\ \dot{\sigma}_j)^2 - (\sum_{j=1}^{K} \log\ \dot{\sigma}_j)^2]}} \tag{14}$$

$$SD_D = 2.3026\,(SD_\beta)(10^{\beta}) \tag{15}$$

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:

$$CV_n\,(\%) = \frac{100\,(SD_n)}{n} \tag{16}$$

$$CV_D\,(\%) = \frac{100\,(SD_D)}{D} \tag{17}$$

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

NOTE 15—For a better representation of SCG behavior of the material, it is recommended that the estimated regression line (that is the "flexural strength-stress rate curve") 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. 2.

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 Type and dimensions of the test specimens,

10.1.3 All relevant material data including vintage data or billet identification data. (Did all specimens come from one billet?) As a minimum, the date the material was manufactured must be reported,

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

10.1.5 Heat treatments or heat exposures, if any,

10.1.6 Methods of specimen cleaning and storage,

10.1.7 All preconditioning (8.5) of specimens prior to testing, if any,

10.1.8 Type, configuration, and material of the test fixture,

10.1.9 Type and configuration of the data acquisition system,

10.1.10 Type of test environment, its conditions, and application method,

10.1.11 Ambient conditions such as temperature and humidity,

10.1.12 Type and configuration of the test machine including the load cell,

10.1.13 Method and magnitude of preloading for each specimen, if any, and

10.1.14 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 test specimens (for example fracture in the gage length) as well as of the invalid test specimens (for example fracture outside the gage length) at each test rate.

10.2.2 Actual loading and stress rates of each specimen to three significant figures.

10.2.3 Strength of every specimen in units of MPa to three significant figures.

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

10.2.5 Graphical representation of test results showing log (flexural strength) as a function of log (stress rate) using all data points, as shown in Fig. 2. Include the determined best-fit, linear regression line in the figure.

10.2.6 Slow crack growth parameters *n* and *D*, and their standard deviations (*SD*s) and coefficients of variation (*CV*s).

10.2.7 Any pertinent fractography information including type, location, and size of fracture origin as well as the degree of SCG, if possible. Also report fracture location relative to the gage section midpoint.

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 specimen to 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 **(4)** and described in term of precision and bias in Section 11 of Test Method C 1161.

11.3 The statistical reproducibility of SCG parameters determined from the constant stress-rate testing has been analyzed in detail (Ref **(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 the given number and range of stress rates, the reproducibility is sensitive to the SCG parameter *n*, Weibull modulus and the number of specimens per test rate, particularly when a high degree of reproducibility is required. 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.

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

12. Keywords

12.1 advanced ceramics; constant stress-rate testing; 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, 13))

X1.1 For most ceramics and glasses, slow crack growth rate can be approximated by the empirical power-law relation (Refs **(14,15)**):

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

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 loading.

For a uniform remote applied stress σ (mode I), the stress intensity factor can be expressed as:

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

where:

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

Using Eq X1.1 and Eq 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:

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

where:

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

is a material/environment parameter.

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

$$\sigma_f^{n+1} = B\,(n+1)\,\sigma_i^{n-2}\,\dot{\sigma} \qquad \text{(X1.4)}$$

In deriving Eq X1.4, it was assumed that $(\sigma_f/\sigma_i)^{n-2} << 1$ since $n \geq 5$ for most ceramics. now taking logarithm for both sides of Eq X1.4 yields:

$$log\ \sigma_f = \frac{1}{n+1}\,log\ \dot{\sigma} + log\ D \qquad \text{(X1.5)}$$

where:

$$log\ D = \frac{1}{n+1}\,log\,[B\,(n+1)\,\sigma_i^{n-2}] \qquad \text{(X1.6)}$$

Therefore, the slow crack growth parameters n and D can be determined by a linear regression analysis based on Eq X1.5 when log (flexural strength) is plotted as a function of log (stress rate).

REFERENCES

(**1**) Ritter, J. E., "Engineering Design and Fatigue Failure of Brittle Materials," *Fracture Mechanics of Ceramics*, Vol 4, New York, 1978, pp. 661-686.

(**2**) Ritter, J. E., Bandyopadhyay, N., and Jakus, K., "Statistical Reproducibility of the Dynamic and Static Fatigue Experiments," *Ceramic Bulletin*, Vol 60, No. 8, 1981, pp. 798-806

(**3**) Jakus, K., Coyne, D. C., and Ritter, J. E., "Analysis of Fatigue Data for Lifetime Prediction for Ceramic Materials," *Journal of Materials Science*, Vol 13, 1978, pp. 2071-2080.

(**4**) Baratta, F. L., Quinn, G. D., and Matthews, W. T.," Errors Associated with Flexure Testing of Brittle Materials," *MTL TR 87-35*, U.S. Army, July 1987.

(**5**) Quinn, G. D., Baratta, F. I., and Conway, J. A., "Commentary on U. S. Army Standard Test Method of Flexure Strength of High Performance Ceramics at Ambient Temperature," *AMMRC 85-21*, U. S. Army, August 1985.

(**6**) Fuller, E. R. and Thomson, R. M.," Lattice Theories of Fracture," *Fracture Mechanics of Ceramics*, Vol 4, New York, 1978, pp. 507-548.

(**7**) Fuller, E. R., Lawn, B. R., and Cook, R. F.," Theory of Fatigue for Brittle Flaws Originating from Residual Stress Concentrations," *Journal of the American Ceramic Society*, Vol 66, No. 5, 1983, pp. 314-321.

(**8**) Choi, S. R. and Salem, J. A., "Inert' Strength of Silicon Nitride Ceramics at Elevated Temperatures," *Ceramic Engineering and Science Proceedings*, Vol 17, No. 3, 1996, pp. 454-461.

(**9**) Dabbs, T. P., Lawn, B. R., and Kelly, P. L., "A Dynamic Fatigue Study of Soda-Lime and Borosilicate Glasses Using Small Scale Indentation Flaws," *Physics and Chemistry of Glasses*, Vol 23, No. 2, 1982, pp. 58-66.

(**10**) Freiman, S. W. and Fuller, E. R., *Interlaboratory Round Robin on Environmental Crack Growth Parameters*, VAMAS unpublished work, 1988.

(**11**) Choi, S. R. and Salem, J. A., "Effect of Preloading on Fatigue Strength in Dynamic Fatigue Testing of Ceramic Materials at Elevated Temperatures," *Ceramic Engineering and Science Proceedings*, Vol 16, 1995, pp. 87-94.

(**12**) Choi, S. R., and Gyekenyesi, J. P., "Fatigue Strength as a Function of Preloading in Dynamic Fatigue Testing of Glass and Ceramics," *ASME Journal of Engineering for Gas Turbines and Power*, Vol 119, 1997, pp.493–499

(**13**) Beck, J. V., and Arnold, K. J., *Parameter Estimation in Engineering and Science*, John Wiley & Sons, Inc., 1977, New York, NY, pp. 135-153.

(**14**) Evans, A. G., "Slow Crack Growth in Brittle Materials under Dynamic Loading Conditions," *International Journal of Fracture*, Vol 10, 1974, pp. 1699-1705.

(**15**) Wiederhorn, S. M., "Subcritical Crack Growth in Ceramics," *Fracture Mechanics of Ceramics*, Vol 2, New York, 1974, pp. 613-646.

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