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# Standard Test Method for Determination of Slow Crack Growth Parameters of Advanced Ceramics by Constant Stress Rate Strength Testing at Ambient Temperature<sup>1</sup>

This standard is issued under the fixed designation C1368; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers the determination of slow crack growth (SCG) parameters of advanced ceramics by using constant stress rate rectangular beam flexural testing, ring-on-ring biaxial disk flexural testing, or direct tensile strength, in which 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 (1-3)<sup>2</sup> 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 E1823, 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 Terminology E1823).

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

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee C28 on Advanced Ceramics and is the direct responsibility of Subcommittee C28.01 on Mechanical Properties and Performance.

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<sup>2</sup> The boldface numbers in parentheses refer to the list of references at the end of this standard.

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>3</sup>

- C1145 Terminology of Advanced Ceramics
- C1161 Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature
- C1239 Practice for Reporting Uniaxial Strength Data and Estimating Weibull Distribution Parameters for Advanced Ceramics
- C1273 Test Method for Tensile Strength of Monolithic Advanced Ceramics at Ambient Temperatures
- C1322 Practice for Fractography and Characterization of Fracture Origins in Advanced Ceramics
- C1499 Test Method for Monotonic Equibiaxial Flexural Strength of Advanced Ceramics at Ambient Temperature
- E4 Practices for Force Verification of Testing Machines
- E6 Terminology Relating to Methods of Mechanical Testing
- E337 Test Method for Measuring Humidity with a Psychrometer (the Measurement of Wet- and Dry-Bulb Temperatures)
- E1823 Terminology Relating to Fatigue and Fracture Testing
- IEEE/ASTM SI 10 American National Standard for Use of the International System of Units (SI): The Modern Metric System

## 3. Terminology

3.1 *Definitions:*

3.1.1 The terms described in Terminologies C1145, E6, and E1823 are applicable to this test method. Specific terms relevant to this test method are as follows:

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

3.1.3 *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.

<sup>3</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard’s Document Summary page on the ASTM website.

3.1.4 *environment, n*—the aggregate of chemical species and energy that surrounds a test specimen. **(E1823)**

3.1.5 *environmental chamber, n*—the container of bulk volume surrounding a test specimen. **(E1823)**

3.1.6 *equibiaxial flexural strength,  $[F/L^2]$ , n*—the maximum stress that a material is capable of sustaining when subjected to flexure between two concentric rings.

3.1.6.1 *Discussion*—This mode of flexure is a cupping of the circular plate caused by loading at the inner load ring and outer support ring. The equibiaxial flexural strength is calculated from the maximum load of a biaxial test carried to rupture, the original dimensions of the test specimen, and Poisson's ratio. **(C1499)**

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

3.1.8 *fracture toughness, n*—a generic term for measures of resistance to extension of a crack. **(E1823)**

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

3.1.9.1 *Discussion*—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 *strength-stress rate curve, n*—a curve fitted to the values of strength at each of several stress rates, based on the relationship between strength and stress rate:  $\log \sigma_f = 1/(n + 1) \log \dot{\sigma} + \log D$ . (See **Appendix X1**.)

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

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

3.1.13 *stress intensity factor,  $K_f$ , n*—the magnitude of the ideal-crack-tip stress field (stress-field singularity) subjected to mode I loading in a homogeneous, linear elastic body. **(E1823)**

3.1.14 *tensile strength,  $S_w$   $[F/L^2]$ , n*—the maximum tensile stress which a material is capable of sustaining.

3.1.14.1 *Discussion*—Tensile strength is calculated from the maximum force during a tension test carried to rupture and the original cross-sectional area of the specimen. **(C1273)**

## 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-stress rate equation, which represent the degree of slow crack growth susceptibility of a material. (See **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. The conventional analysis of constant stress rate testing is based on a number of critical assumptions, the most important of which are listed in the next paragraphs.

4.2 The flexural stress computation for the rectangular beam test specimens or the equibiaxial disk flexure test specimens 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 test specimen sizes and fixtures for rectangular beam test specimens should be in accordance with Test Method **C1161**, which provides a balance between practical configurations and resulting errors, as discussed in Refs (4, 5). Only four-point test configuration is allowed in this test method for rectangular beam specimens. Three-point test configurations are not permitted. The test specimen sizes and fixtures for disk test specimens tested in ring-on-ring flexure should be chosen in accordance with Test Method **C1499**. The test specimens for direct tension strength testing should be chosen in accordance with Test Method **C1273**.

4.4 The SCG parameters ( $n$  and  $D$ ) are determined by fitting the measured experimental data to a mathematical relationship between 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 3—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 (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 strength and stress rate was derived based on the assumption that the slow crack growth parameter is at least  $n \geq 5$  (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 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 (2). Guidelines are provided in this test method.

NOTE 4—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 C1322).

4.10 The conventional analysis of constant stress rate testing is based on a critical assumption that stress is uniform throughout the test piece. This is most easily achieved in direct tension test specimens. Only test specimens that fracture in the inner gauge section in four-point testing should be used. Three-point flexure shall not be used. Breakages between the outer and inner fixture contact points should be discounted. The same requirement applies to biaxial disk strength testing. Only fractures which occur in the inner loading circle should be used. Furthermore, it is assumed that the fracture origins are near to the tensile surface and do not grow very large relative to the thickness of rectangular beam flexure or disk strength test specimens.

4.11 The conventional analysis of constant stress rate testing is also based on a critical assumption that the same type flaw controls strength in all specimens at all loading rates. If the flaw distribution is multimodal, then the conventional analysis in this standard may produce erroneous slow crack growth parameter estimates.

## 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 (strength) and log (applied stress rate) (see Appendix X1) may start to deviate at a certain high stress rate at which slow crack growth dimin-

ishes 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 (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 be  $\leq 2000$  MPa/s.

NOTE 5—The strength plateau of a material can be checked by measuring an inert strength in an appropriate inert medium.

NOTE 6—When testing in environments with less than 100 % concentration of the corrosive medium (for example, air), the use of stress rates greater than  $\sim 1$  MPa/s can result in significant errors in the slow crack growth parameters due to averaging of the regions of the slow crack growth curve (9). Such errors can be avoided by testing in 100 % concentration of the corrosive medium (for example, in water instead of humid air). For the case of 100 % concentration of the corrosive medium, stress rates as large as  $\sim 2000$  MPa/s may be acceptable.

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 E4. 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 strength shall be accurate to within  $\pm 1.0$  % at any load within the selected load rate and load range of the testing machine as defined in Practices E4. 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, Four-Point Rectangular Beam Flexure*—The configurations and mechanical properties of test fixtures should be in accordance with Test Method C1161. 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 7—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- $\frac{1}{4}$ -point fixture configuration as described in 6.2 of Test Method **C1161** shall be used in this test method. Three-point flexure is not permitted. 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 *Test Fixtures, Equibiaxial Disk Flexural Strength*—The configurations and mechanical properties of test fixtures should be in accordance with Test Method **C1499**. 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. See **Note 7**. The test fixtures shall be stiffer than the specimen, so that most of the crosshead or actuator travel is imposed onto the specimen.

6.4 *Test Fixtures, Tensile Strength*—The configurations and mechanical properties of test fixtures should be in accordance with Test Method **C1273**. 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. See **Note 7**. The test fixtures shall be stiffer than the specimen, so that most of the crosshead or actuator travel is imposed onto the specimen.

6.5 *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 vice versa.

6.6 *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 replenish 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 flexure specimens as described in 7.1 of Test Method **C1161** shall be used in this test method. The types and

dimensions of disk-shaped flexure specimens as described in 8.1 of Test Method **C1499** shall be used in this test method. The types and dimensions of tension strength specimens as described in 8.1 of Test Method **C1273** shall be used in this test method.

7.2 *Specimen Preparation*—Specimen fabrication and preparation methods as described in the appropriate sections of Test Method **C1161**, **C1273**, or **C1499** 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 ten 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 8**—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 in Test Method **C1161**, **C1273**, or **C1499**. For example, for four-point flexural strength of rectangular beam specimens see Section 6 of Test Method **C1161**. 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 affect the statistical reproducibility of SCG parameters but depend 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 9—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 (10, 11). 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 (10) 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 (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 rectangular beam or biaxial disk test specimens beyond the outer bearing cylinders or the outer support ring, respectively.

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 test fixture and the specimen to ensure even loading of the rectangular beam or biaxial disk test specimens. If uneven 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 gauge section of 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 E337. It is recommended that 100 % concentration of the corrosive medium be used in order to minimize averaging of the fatigue curve regions and thereby allow the use of stress rates greater than 1 MPa/s (9). An example of this is the use of water instead of humid air.

NOTE 10—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.

NOTE 11—Preloads truncate the slow crack curve and can result in errors in the estimated slow crack growth parameters. Testing in 100 % concentration of the corrosive medium extends the region of the slow crack growth curve for which the model used in this standard is applicable, and thereby allows preloads (9). When in doubt it is recommended that preloads greater than that required for setup not be used (see 8.4).

8.6.2 It has been shown that in constant stress rate testing, considerably high preloading can be applied to ceramic specimens with no change in the strength obtained, resulting in a significant reduction of test time (12, 13). The relationship between strength and preloading is as follows:

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

where:

- $\sigma^*$  = normalized strength =  $\sigma_{fp}/\sigma_{fn}$ ,
- $\alpha_p$  = preloading factor ( $0 \leq \alpha_p < 1.0$ ) =  $\sigma_o/\sigma_{fn}$ ,
- $\sigma_{fp}$  = strength with preloading,

$\sigma_{fn}$  = strength without preloading,  
 $\sigma_o$  = preload stress, and  
 $n$  = slow crack growth parameter.

8.6.3 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, 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 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 (12, 13). 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.

8.6.4 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 6.5 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 9.

8.8 When tests are conducted in ambient air, put cotton, crumbled tissues, or other appropriate material around the flexural strength or equibiaxial strength 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  $\pm 1.0$  %.

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 dimensions of each test specimen in accordance with Test Method C1161 for flexural strength test specimens, Test Method C1499 for equibiaxial disk strength specimens, or Test Method C1273 for tensile strength specimens. 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.

8.10.4 Note that the specimens broken outside the gauge length (the inner span region for the rectangular beam specimens, or the inner loading circle in equibiaxial disk specimens, or the middle gauge section in tension specimens) should not be used in determining the SCG parameters. Results from the specimens broken outside the gauge 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 gauge length. Flaws located in the reduced-stress regions of a test piece may grow at different rates than flaws located in the inner gauge sections even if they have comparable initial stress intensities. 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 gauge 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 C1239.

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 C1322.

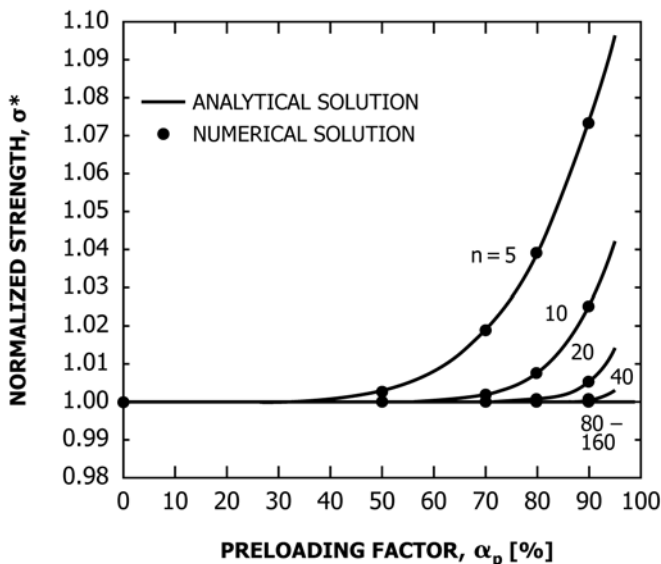


FIG. 1 Normalized Strength as a Function of Preloading for Different Slow Crack Growth Parameter  $n$ 's (12)