Designation: C1368 – 10 (Reapproved 2017)

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

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, or 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)[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 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

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, predominantly nonmetallic, inorganic, ceramic material having specific functional attributes. **(C1145)**

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

A Summary of Changes section appears at the end of this standard

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.

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, 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.

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

3.1.9 *inert strength, 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_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. **(E1823)**

3.1.14 *tensile strength [F/L$_2$], n—S$_u$*—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 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 (**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 ≤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 ~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 ~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 within ±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 ¼-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 vise 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 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 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 Methods 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 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 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 Methods 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 (≥ 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 estmated 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:

$$\sigma^* = \left(1 + \alpha_p^{n+1}\right)^{\frac{1}{n+1}} \qquad (1)$$

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.

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 % (= α_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 (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.
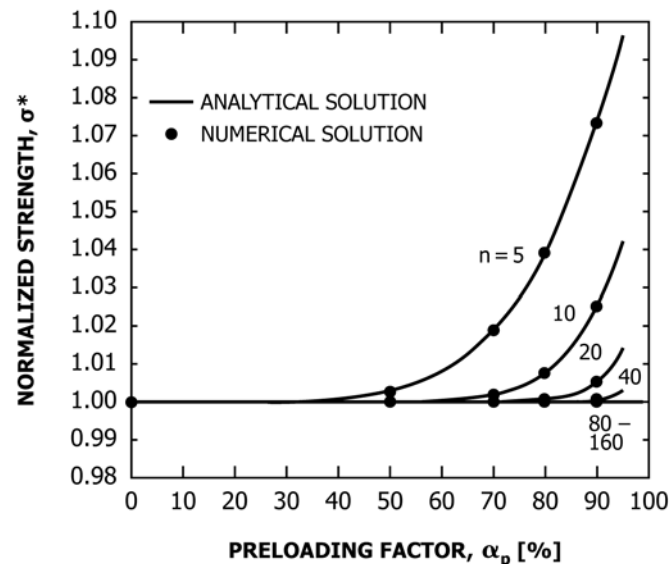
8.7 For either load-controlled or displacement-control mode, record a load-versus-time curve for each test in order to determine the actual loading rate and thus to calculate the corresponding stress rate (see also 6.5 and 9.2). The actual loading rate in units of newtons per second should be determined 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 ±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.

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

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.

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 Compute the flexural strength, or the equibiaxial flexural strength, or the tensile strength in accordance with the formulas in Test Methods C1161, C1273, or C1499.

9.1.2 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:

$$\bar{\sigma}_f = \frac{\sum_{j=1}^{N} \sigma_j}{N} \quad (2)$$

$$SD_\sigma = \sqrt{\frac{\sum_{j=1}^{N} (\sigma_j - \bar{\sigma}_f)^2}{N-1}} \quad (3)$$

$$CV_\sigma (\%) = \frac{100 (SD_\sigma)}{\bar{\sigma}_f} \quad (4)$$

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

9.2 *Stress Rate:*

9.2.1 The stress rate of each rectangular beam flexure specimen in four-point ¼-point loading subjected to either displacement-controlled or load-controlled mode is calculated using the actual loading rate determined (8.7) as follows:

$$\dot{\sigma} = \frac{3\dot{P}L}{4bd^2} \quad (5)$$

where:
$\dot{\sigma}$ = stress rate, MPa/s,
$\dot{P}$ = loading rate, N/s,
L = outer (support) span of the test fixture, mm,
b = specimen width, mm, and
d = specimen thickness, mm.

9.2.2 The stress rate of each equibiaxial disk flexure specimen subjected to either displacement-controlled or load-controlled mode is calculated in accordance with 9.2.3 of Test Method C1499.

9.2.3 The stress rate of each tension specimen subjected to either displacement-controlled or load-controlled mode is calculated in accordance with 9.3.2 of Test Method C1273.

9.2.4 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 strength-stress rate diagram), as shown in Fig. 2. The SCG parameters n and D can be determined by a linear regression analysis (14) 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):
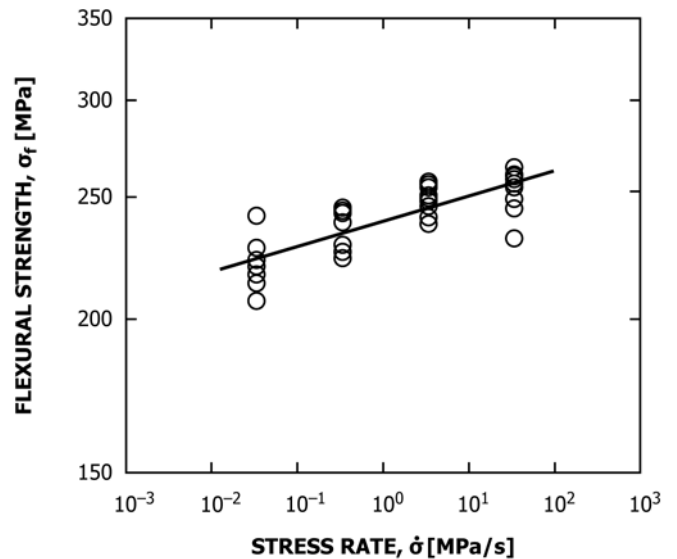
$$\log \sigma_f = \frac{1}{n+1}\log \dot{\sigma} + \log D \quad (6)$$

NOTE 12—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 13—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:

$$\alpha = \frac{K\sum_{j=1}^{K} (\log \dot{\sigma}_j \log\sigma_j) - \left(\sum_{j=1}^{K}\log \dot{\sigma}_j \sum_{j=1}^{K}\log \sigma_j\right)}{K\sum_{j=1}^{K} (\log \dot{\sigma}_j)^2 - \left(\sum_{j=1}^{K}\log \dot{\sigma}_j\right)^2} \quad (7)$$

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)

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:

$$n = \frac{1}{\alpha} - 1 \qquad (8)$$

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

$$\beta = \frac{\left(\sum_{j=1}^{K} \log \sigma_j\right) \sum_{j=1}^{K} (\log \dot{\sigma}_j)^2 - \left(\sum_{j=1}^{K} \log \dot{\sigma}_j \log \sigma_j\right)\left(\sum_{j=1}^{K} \log \dot{\sigma}_j\right)}{K \sum_{j=1}^{K} (\log \dot{\sigma}_j)^2 - \left(\sum_{j=1}^{K} \log \dot{\sigma}_j\right)^2} \qquad (9)$$

where:

β = intercept.

9.3.1.4 Calculate the SCG parameter D as follows:

$$D = 10^\beta \qquad (10)$$

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

$$SD_\alpha = \sqrt{\frac{K}{K-2} \frac{\sum_{j=1}^{K} (\alpha \log \dot{\sigma}_j + \beta - \log \sigma_j)^2}{K \sum_{j=1}^{K} (\log \dot{\sigma}_j)^2 - \left(\sum_{j=1}^{K} \log \dot{\sigma}_j\right)^2}} \qquad (11)$$

$$SD_n = \frac{SD_\alpha}{\alpha^2} \qquad (12)$$

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:

$$SD_\beta = \sqrt{\frac{\sum_{j=1}^{K} (\alpha \log \dot{\sigma}_j + \beta - \log \sigma_j)^2 \sum_{j=1}^{K} (\log \dot{\sigma}_j)^2}{(K-2)\left[K \sum_{j=1}^{K} (\log \dot{\sigma}_j)^2 - \left(\sum_{j=1}^{K} \log \dot{\sigma}_j\right)^2\right]}} \qquad (13)$$

$$SD_D = 2.3026 \, (SD_\beta)(10^\beta) \qquad (14)$$

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:

$$CV_n \, (\%) = \frac{100 \, (SD_n)}{n} \qquad (15)$$

$$CV_D \, (\%) = \frac{100 \, (SD_D)}{D} \qquad (16)$$

where:

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

NOTE 14—For a better representation of SCG behavior of the material, it is recommended that the estimated regression line (that is the "strength-

stress rate curve") be included in the 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 gauge length) as well as of the invalid test specimens (for example fracture outside the gauge 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 (9.1.2).

10.2.5 Graphical representation of test results showing log (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 (SDs) and coefficients of variation (CVs).

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 gauge section midpoint.

11. Precision and Bias

11.1 The 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 C1239. 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 of rectangular beam flexure test specimens have been analyzed in detail in Ref (4) and described in term of precision and bias in Section 11 of Test Method C1161.

11.3 The statistical reproducibility of SCG 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 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; biaxial flexure; constant stress-rate testing; equibiaxial flexural strength; flexural strength; flexural testing; four-point flexure; slow crack growth; slow crack growth parameters; tensile strength

APPENDIX

(Nonmandatory Information)

X1. DERIVATION OF STRENGTH AS A FUNCTION OF APPLIED STRESS RATE IN CONSTANT STRESS-RATE TESTING (DYNAMIC FATIGUE EQUATION) (1, 14)

X1.1 For most ceramics and glasses, slow crack growth rate can be approximated by the empirical power-law relation (15,16):

$$v = \frac{da}{dt} = AK_I^n = A*\left(\frac{K_I}{K_{IC}}\right)^n \qquad \text{(X1.1)}$$

where:

v = slow crack growth rate,
a = crack length,
t = time,
$A, A*,$ 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:

$$K_I = Y\sigma\sqrt{a} \qquad \text{(X1.2)}$$

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:

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

where:

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

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 (strength) is plotted as a function of log (stress rate).

X1.2 For life prediction of ceramic components the slow crack growth parameters B or A of Eq X1.1 and Eq X1.4 are needed. The parameters can be calculated in terms of the slope and intercept from (17):

$$B = \frac{\alpha(10^{\beta/\alpha})}{\sigma_i^{\left(\frac{1}{\alpha}-3\right)}} \qquad \text{(X1.7)}$$

$$A* = \frac{2K_{Ic}^2\sigma_i^{\left(\frac{1}{\alpha}-3\right)}}{10^{\beta/\alpha}(1-3\alpha)Y^2} = \frac{2K_{Ic}^2}{B(n-2)Y^2} \qquad \text{(X1.8)}$$

The associated standard deviations need to be calculated as logarithms of the parameters for accuracy:

9

$$SD_{lnB} \approx \frac{1}{\alpha} \sqrt{ \begin{array}{c} Q^2 \dfrac{SD_\alpha^2}{\alpha^2} + (ln10)^2\, SD_\beta^2 \\[2mm] + (1-3\alpha)^2\, SD_{ln\sigma_i}^2 + 2Q\,ln10\, \dfrac{Cov(\alpha,\beta)}{\alpha} \end{array} } \quad (X1.9)$$

$$SD_{lnA^*} \approx \frac{1}{\alpha} \sqrt{ \begin{array}{c} 4\alpha^2 \dfrac{SD_{K_{Ic}}^2}{K_{Ic}^2} + \left(Q - \dfrac{\alpha}{1-3\alpha}\right)^2 \dfrac{SD_\alpha^2}{\alpha^2} + (ln10)^2\, SD_\beta^2 \\[2mm] + (1-3\alpha)^2\, SD_{ln\sigma_i}^2 + 2\,ln10\left(Q - \dfrac{\alpha}{1-3\alpha}\right) \dfrac{Cov(\alpha,\beta)}{\alpha} \end{array} } \quad (X1.10)$$

where:

$$Q = \alpha - \beta\, ln10 + ln\sigma_i$$

and

$$Cov(\alpha,\beta) = -SD_\alpha^2 \left(\overline{log\,\dot\sigma}\right) \quad (X1.11)$$

where $\overline{log\,\dot\sigma}$ is the mean of the log of the stressing rates applied. Probability limits on the parameters B and A can be calculated from:

$$B_{Upper \atop Lower} = EXP\left[ln\,B \pm t(SD_{ln\,B})\right] \quad \text{and} \quad A_{Upper \atop Lower} = EXP\left[lnA \pm t(SD_{ln\,A})\right] \quad (X1.12)$$

by using Student's t distribution for the DOF and probability level desired. If the DOF (degree-of-freedom) are greater than ~40, then:

$$B_{Upper \atop Lower} = EXP\left[ln\,B \pm l(SD_{ln\,B})\right] \quad \text{and} \quad A_{Upper \atop Lower} = EXP\left[ln\,A \pm l(SD_{ln\,A})\right] \quad (X1.13)$$

where l is the number of standard deviations corresponding to the probability level desired. The DOF, φ, is given by:

$$\frac{(SD_{ln\,B}^2)^2}{\varphi_{ln\,B}} = \frac{1}{\varphi_{ln\,\sigma_i}}\left[\frac{(1-3\alpha)^2}{\alpha^2}\, SD_{ln\,\sigma_i}^2\right]^2 \quad (X1.14)$$

$$+ \frac{1}{\varphi_{\alpha\beta}}\left[Q^2 \frac{SD_\alpha^2}{\alpha^4} + (ln10)^2 \frac{SD_\beta^2}{\alpha^2} + 2Q\,ln10\, \frac{Cov(\alpha,\beta)}{\alpha^3}\right]^2$$

and

$$\frac{(SD_{ln\,A}^2)^2}{\varphi_{ln\,A}} = \frac{1}{\varphi_{ln\,K_{Ic}}}\left(4SD_{lnK_{Ic}}^2\right)^2 + \frac{1}{\varphi_{ln\,\sigma_i}}\left[\frac{(1-3\alpha)^2}{\alpha^2}\, SD_{ln\,\sigma_i}^2\right]^2 \quad (X1.15)$$

$$+ \frac{1}{\varphi_{\alpha\beta}}\left[\left(Q - \frac{\alpha}{1-3\alpha}\right)^2 \frac{SD_\alpha^2}{\alpha^4} + (ln\,10)^2 \frac{SD_\beta^2}{\alpha^2} + 2\,ln\,10\left(Q - \frac{\alpha}{1-3\alpha}\right) \frac{Cov(\alpha,\beta)}{\alpha^3}\right]^2$$

where φ_{σ_i} is the DOF in inert strength (number of inert strength tests minus one) and $\varphi_{\alpha\beta}$ is the DOF in regression (number of constant stress rate tests minus two).

When the velocity equation is written as:

$$v = AK_I^n \quad (X1.16)$$

the values of A and $SD_{ln\,A}$ can be estimated from:

$$A = \frac{2K_{Ic}^{\left(3-\frac{1}{\alpha}\right)} \sigma_i^{\left(\frac{1}{\alpha}-3\right)}}{10^{\beta/\alpha}(1-3\alpha)Y^2} = \frac{2K_{Ic}^{2-n}}{B(n-2)Y^2} \quad (X1.17)$$

$$SD_{ln\,A} \approx \frac{1}{\alpha} \sqrt{ \begin{array}{c} (3\alpha - 1)^2 \dfrac{SD_{K_{Ic}}^2}{K_{Ic}^2} + \left(Q - \dfrac{\alpha}{1-3\alpha} - ln\,K_{Ic}\right)^2 \dfrac{SD_\alpha^2}{\alpha^2} \\[2mm] + (ln\,10)^2\, SD_\beta^2 + (1-3\alpha)^2\, SD_{ln\,\sigma_i}^2 \\[2mm] + 2\,ln10\left(Q - \dfrac{\alpha}{1-3\alpha} - ln\,K_{Ic}\right) \dfrac{Cov(\alpha,\beta)}{\alpha} \end{array} } \quad (X1.18)$$

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SUMMARY OF CHANGES

Committee C28 has identified the location of selected changes to this standard since the last issue (C1368 – 06) that may impact the use of this standard. (Approved Dec. 1, 2010.)

(1) The standard is expanded to include direct tensile strength testing in accordance with Test Method C1273.

(2) The standard is expanded to include equibiaxial flexural strength testing in accordance with Test Method C1499.

(3) The standard explicitly states that three-point flexure is not permitted for rectangular beam flexural strength specimens.