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.)

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[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.

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 (≥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