



Designation: C1576 – 05

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

This standard is issued under the fixed designation C1576; 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 flexural testing in which time to failure of flexure test specimens is determined in *four-point* flexure as a function of constant applied stress in a given environment at ambient temperature. In addition, test specimen fabrication methods, test stress levels, data collection and analysis, and reporting procedures are addressed. The decrease in time to failure with increasing applied stress in a specified environment is the basis of this test method that enables the evaluation of slow crack growth parameters of a material. The preferred analysis in the present method is based on a power law relationship between crack velocity and applied stress intensity; alternative analysis approaches are also discussed for situations where the power law relationship is not applicable.

NOTE 1—The method in this test method is frequently referred to as static *fatigue* or stress-rupture 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 that occurs exclusively under cyclic loading, as defined in Terminology E1823, this test method uses the term constant stress testing rather than static *fatigue* testing.

1.2 This test method applies primarily to monolithic advanced ceramics that are macroscopically homogeneous and isotropic. This test method may also be applied to certain whisker- or particle-reinforced ceramics as well as certain discontinuous fiber-reinforced composite ceramics that exhibit macroscopically homogeneous behavior. Generally, continuous fiber ceramic composites do not exhibit macroscopically isotropic, homogeneous, continuous behavior, and the application of this test method to these materials is not recommended.

1.3 This test method is intended for use with various test environments such as air, other gaseous environments and liquids.

1.4 The values stated in SI units are to be regarded as the standard and in accordance with SI10-02 IEEE/ASTM SI 10 .

1.5 *This test method may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety concerns 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:<sup>3</sup>

- C1145 Terminology of Advanced Ceramics
- C1161 Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature
- C1322 Practice for Fractography and Characterization of Fracture Origins in Advanced Ceramics
- C1368 Test Method for Determination of Slow Crack Growth Parameters of Advanced Ceramics by Constant Stress-Rate Flexural Testing at Ambient Temperature
- C1465 Test Method for Determination of Slow Crack Growth Parameters of Advanced Ceramics by Constant Stress-Rate Flexural Testing at Elevated Temperatures
- E4 Practices for Force Verification of Testing Machines
- E6 Terminology Relating to Methods of Mechanical Testing
- E112 Test Methods for Determining Average Grain Size
- E337 Test Method for Measuring Humidity with a Psychrometer (the Measurement of Wet- and Dry-Bulb Temperatures)
- E399 Test Method for Linear-Elastic Plane-Strain Fracture Toughness  $K_{Ic}$  of Metallic Materials
- E1823 Terminology Relating to Fatigue and Fracture Testing

<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.

Current edition approved June 1, 2005. Published June 2005 DOI: 10.1520/C1576-05.

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

<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. Terminology

3.1 *Definitions*: 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:

3.1.1 *advanced ceramic,  $n$* —highly engineered, high performance, predominately non-metallic, inorganic, ceramic material having specific functional attributes. **C1145**

3.1.2 *constant applied stress,  $\sigma [FL^{-2}]$* ,  $n$ —constant maximum flexural stress applied to a specified beam test specimen by using a constant static force with a test machine or a test fixture.

3.1.3 *constant applied stress-time to failure diagram*—plot of constant applied stress against time to failure. Constant applied stress and time to failure are both plotted on logarithmic scales.

3.1.4 *constant applied stress-time to failure curve*—curve fitted to the values of time to failure at each of several applied stresses.

NOTE 2—In the ceramics literature, this is often called a static fatigue curve.

3.1.5 *test environment,  $n$* —aggregate of chemical species and energy that surrounds a test specimen. **E1823**

3.1.6 *test environmental chamber,  $n$* —container surrounding the test specimen that is capable of providing controlled local environmental condition. **C1368, C1465**

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

3.1.8 *fracture toughness (critical stress intensity factor)  $K_{IC} [FL^{-3/2}]$* ,  $n$ —generic term for measures of resistance to extension of a crack. **E1823, E399**

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

NOTE 3—An inert condition may be obtained by using vacuum, low temperature, very fast test rate, or an inert environment such as silicone oil or high purity dry  $N_2$ .

3.1.10 *R-curve,  $n$* —plot of crack-extension resistance as a function of stable crack extension. **C1145**

3.1.11 *run-out,  $n$* —test specimen that does not fail before a prescribed test time.

3.1.12 *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. **C1368, C1465**

3.1.13 *slow crack growth (SCG) parameters*—parameters estimated as constants in the log (*time to failure*) versus log (*constant applied stress*), which represent a measure of susceptibility to slow crack growth of a material (see **Appendix X1**).

3.1.14 *stress intensity factor,  $K_I [FL^{-3/2}]$* ,  $n$ —magnitude of the ideal-crack-tip stress field stress field singularity) subjected to mode I loading in a homogeneous, linear elastic body. **E1823**

### 4. Significance and Use

4.1 The service life of many structural ceramic components is often limited by the subcritical growth of cracks. This test method provides an approach for appraising the relative slow crack growth susceptibility of ceramic materials under specified environments at ambient temperature. Furthermore, this test method may establish the influences of processing variables and composition on slow crack growth as well as on strength behavior of newly developed or existing materials, thus allowing tailoring and optimizing material processing for further modification. In summary, this test method may be used for material development, quality control, characterization, design code or model verification, and limited design data generation purposes.

NOTE 4—Data generated by this test method do not necessarily correspond to crack velocities that may be encountered in service conditions. The use of data generated by this test method for design purposes, depending on the range and magnitude of applied stresses used, may entail extrapolation and uncertainty.

4.2 This test method is related to Test Method **C1368** (constant stress-rate flexural testing); however, Test Method **C1368** uses constant stress rates to determine corresponding flexural strengths whereas this test method employs constant stress to determine corresponding times to failure. In general, the data generated by this test method may be more representative of actual service conditions as compared with those by constant stress-rate testing. However, in terms of test time, constant stress testing is inherently and significantly more time-consuming than constant stress rate testing.

4.3 The flexural stress computation in this test method is based on simple elastic 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 grain size should be no greater than one-fiftieth ( $1/50$ ) of the beam depth as measured by the mean linear intercept method (Test Methods **E112**). In cases where the material grain size is bimodal or the grain size distribution is wide, the limit should apply to the larger grains.

4.4 The test specimen sizes and test fixtures have been selected in accordance with Test Methods **C1161** and **C1368**, which provides a balance between practical configurations and resulting errors, as discussed in Refs (**4,5**).

4.5 The data are evaluated by regression of log applied stress vs. log time to failure to the experimental data. The recommendation is to determine the slow crack growth parameters by applying the power law crack velocity function. For derivation of this, and for alternative crack velocity functions, see **Appendix X1**.

NOTE 5—A variety of crack velocity functions exist in the literature. A comparison of the functions for the prediction of long-term static fatigue data from short-term dynamic fatigue data (**6**) indicates that the exponential forms better predict the data than the power-law form. Further, the exponential form has a theoretical basis (**7-10**); however, the power law form is simpler mathematically. Both have been shown to fit short-term test data well.

4.6 The approach used in this test method assumes that the material displays no rising R-curve behavior, that is, no increasing fracture resistance (or crack-extension resistance) with increasing crack length. The existence of such behavior cannot be determined from this test method. The analysis further assumes that the same flaw type controls all times-to-failure.

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

4.8 Like strength, time to failure of advanced ceramics subjected to slow crack growth is probabilistic in nature. Therefore, slow crack growth that is determined from times to failure under given constant applied stresses is also a probabilistic phenomenon. The scatter in time to failure in constant stress testing is much greater than the scatter in strength in constant stress-rate (or any strength) testing (1, 11-13) (see Appendix X2). Hence, a proper range and number of constant applied stresses, in conjunction with an appropriate number of test specimens, are required for statistical reproducibility and reliable design data generation (1-3). This test method provides guidance in this regard.

4.9 The time to failure of a ceramic material for a given test specimen and test fixture configuration is dependent on its inherent resistance to fracture, the presence of flaws, applied stress, and environmental effects. Fractographic analysis to verify the failure mechanisms has proven to be a valuable tool in the analysis of SCG data to verify that the same flaw type is dominant over the entire test range (14, 15), and it is to be used in this test method (see Practice C1322).

## 5. Interferences

5.1 Slow crack growth may be the product of both mechanical and chemical driving forces. The chemical driving force for a given material can vary strongly with the composition and temperature of a test environment. Testing is conducted in environments representative of service conditions so as to evaluate material performance under use conditions. Note that slow crack growth testing, particularly constant stress testing, is very time-consuming. The overall test time is considerably greater in constant stress testing than in constant stress-rate testing. Because of this longer test time, the chemical variables of the test environment must be prevented from changing significantly throughout all test times. Inadequate control of these chemical variables may result in inaccurate time-to-failure data, especially for materials that are more sensitive to the test environment.

5.2 Depending on the degree of SCG susceptibility of a material, the linear relationship between  $\log(\text{constant applied stress})$  and  $\log(\text{time to failure})$  may start to deviate at a certain high applied stress where the crack velocity increases rapidly with a subsequently short test duration, that is, the applied stress approaches the strength, (see Fig. 1). This is analogous to the occurrence of a strength plateau observed at higher test rates in constant stress-rate testing (16). If the time-to-failure

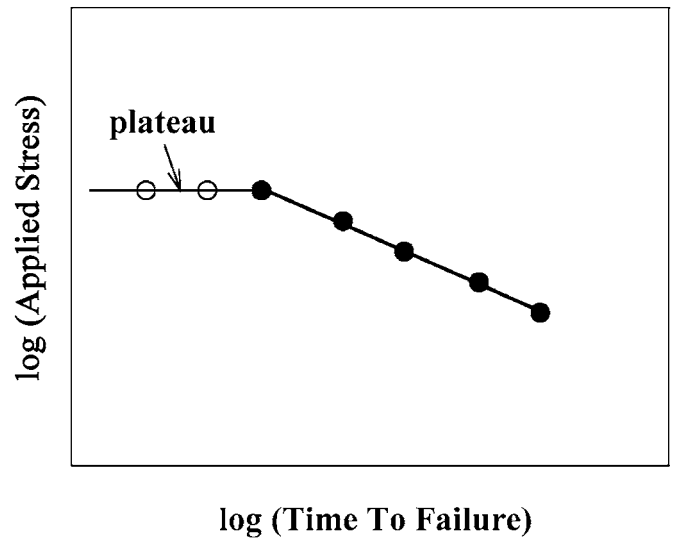


FIG. 1 Schematic Diagram Showing Unacceptable (Average) Data Points (With an “Open” Symbol) in the Plateau Region in Determining Slow Crack Growth (SCG) Parameters

data determined in this plateau region are included in the analysis, a misleading estimate of the SCG parameters will be obtained (17). Therefore, the strength data in the plateau shall be excluded as data points in estimating the SCG parameters of the material. Similarly, a plateau can also exist at the fatigue limit end of the curve, and these data points shall also be excluded in estimating the SCG parameters.

NOTE 6—There are no simple guidelines in determining whether a plateau region is reached; however, with knowledge of the inert strength and the fracture toughness of the test material, the slow crack growth rate – applied stress intensity ( $v$ -K) curve may be determined. Evaluating this will help determine where the experimental conditions fall.

5.3 When testing a material exhibiting a high SCG resistance (typically SCG parameter  $n > 70$ ), an unrealistically large number of test specimens may be required in a small range of applied stresses since a significant number of test specimens may be expected to fail while loading. Furthermore, if lower stresses are to be used, unrealistically long test times are to be expected. As a result, *practical, specific, and quantitative* values of SCG parameters required for life prediction can only with great difficulty be determined for this type of material (18). In this case, a companion test method for constant stress-rate testing, Test Method C1368, may be utilized instead to determine the corresponding SCG parameters of the material. The constant stress-rate test may be used, provided the same flaw types are activated in both stress states.

5.4 Surface preparation of test specimens can introduce flaws that may have pronounced effects on flexural strength and thus time to failure. Machining damage imposed during test 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. It should be understood that the final machining steps may or may not negate machining damage introduced during the earlier course or intermediate machining steps. In some cases, test specimens need to be tested in the as-processed condition



to simulate a specific service condition. Test-specimen fabrication history may play an important role in strength as well as time-to-failure behavior, which consequently may affect the values of the SCG parameters to be determined. Therefore, the test specimen fabrication history shall be reported. In addition the nature of fabrication used for certain advanced ceramic components may require testing of specimens with surfaces in the as-fabricated condition (that is, it may not be possible, desired or required to machine some test specimens directly in contact with test fixture components). In such cases, a fully articulated test fixture is required. However, for very rough or wavy as-fabricated surfaces, eccentricities in the stress state due to non-symmetric cross-sections as well as variations in the cross-sectional dimensions may also interfere with the strength measurement.

5.5 Premature fracture may be initiated at surface flaws (for example, scratches, edge chips) introduced while handling the specimens.

5.6 Fractures that consistently initiate near or just outside the load pins may be due to factors such as friction or contact stresses introduced by the load fixtures or via misalignment of the test specimen load pins. Failure of test specimens initiated consistently from their edges may be due to poor specimen preparation (for example, severe grinding or very poor edge preparation) or excessive twisting stresses at the specimen edges (4, 5, 19).

5.7 Fractures may initiate from different flaw types (for example, surface flaws like scratches and machining flaws, or pores and agglomerates that may be located in the volume or at the surface of the specimens). The analysis performed in this test method assumes that all failures initiate from similar types of flaws as confirmed by fractography in accordance with Practice C1322.

## 6. Apparatus

6.1 *Test Machine*—Dead weight or universal test machines capable of maintaining a constant force may be used for constant stress testing. The variations in the selected force shall not exceed  $\pm 1.0\%$  of the nominal value at any given time during the test. The force must be monitored and the variations in the selected force shall not exceed the  $\pm 1.0\%$  limit at any given time during the test. Test machines used for this test method shall conform to the requirements of Practices E4.

6.2 *Test Fixtures*—The configurations and mechanical properties of test fixtures shall 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 significantly react with or contaminate either the test specimen or the test environment.

**NOTE 7**—For testing in distilled water, for example, it is recommended that the test fixture be fabricated from stainless steel. The bearing cylinders may be machined from hardenable stainless steel (for example, 316 SS) 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 Test Method C1161 shall be used in this test method. Three-point flexure shall not be used.

6.2.2 *Bearing Cylinders*—The requirements of dimensions and mechanical properties of bearing cylinders as described in

Test Method C1161 shall be used in this test method. The bearing cylinders shall be free to roll in order to relieve frictional constraints as described in Test Method C1161.

6.2.3 *Semiarticulating Four-Point Fixture*—The semiarticulating four-point fixture as described in Test Method C1161 may be used in this test method. This fixture shall be used when the parallelism requirements of test specimens are met according to Test Method C1161.

6.2.4 *Fully Articulating Four-Point Fixture*—The fully articulating four-point fixture as described in Test Method C1161 may be used in this test method. Specimens that do not meet the parallelism requirements in Test Method C1161 due to the nature of fabrication process (as-fired, heat-treated, or oxidized), shall be tested in this fully articulating fixture.

6.3 *Environmental Facility*—For testing in an environment other than ambient air, use a chamber that is inert to the test environment, capable of safely containing the environment, and allowing monitoring of environments to ensure consistency. The chamber shall be sufficiently large to immerse the test specimen in the test medium. A circulation or mixing system may be desirable, depending on the conditions to be simulated. Additionally, the facility shall be able to safely contain the test environment. If it is necessary to direct force through bellows, fittings, or seals, it shall be verified that force losses or errors do not exceed 1% of the prospective applied force. If ambient temperature tests are conducted under constant environmental conditions, then control the temperature and relative humidity to within  $\pm 3^\circ\text{C}$  and  $\pm 10\%$  of the set humidity level, respectively.

6.4 *Data Acquisition*—Accurate determination of time to failure (or test time in case of run-out) is important since time to failure is the only dependent variable in this test method. This is particularly important when time to failure is relatively short ( $<10\text{s}$ ) when a higher applied stress is used. Devices to measure time to failure may be either digital or analog and incorporate a switching mechanism to stop the device at test specimen failure. The recording device shall be accurate to within  $\pm 1\%$  of the selected range. If universal test machines are used, at the minimum, an autographic record of applied force versus time shall be determined during testing. Either analog chart recorders or digital data acquisition systems can be used for this purpose. Recording devices shall be accurate to 1.0% of the recording range and shall have a minimum data acquisition rate sufficient to adequately describe the whole test series. The appropriate data acquisition rate depends on the actual time to failure (that is, magnitude of applied stress), but should preferably be in the 0.2 to 50 Hz range (50 Hz for times less than 5 s, 10 Hz for times between 5 s and 10 min, 1 Hz for times between 10 min and 5 h, and 0.2 Hz for times over 5 h).

6.5 *Dimension Measuring Devices*—Micrometers and other devices used for measuring test specimen dimensions shall have a resolution of 0.002 mm or smaller. To avoid damage in the gage section area, depth measurements should be made using a flat, anvil type micrometer. Ball-tipped or sharp anvil micrometers should not be used because localized damage (for example, cracking) can be induced.

## 7. Test Specimen

7.1 *Specimen Size*—The types and dimensions of rectangular beam specimens as described in Test Method C1161 shall be used in this test method.

7.2 *Specimen Preparation*—Specimen fabrication and preparation methods as described in Test Method C1161 shall be used in this test method.

7.3 *Specimen Dimensions*—Determine the width and depth of each test specimen as described in Test Method C1161, either optically or mechanically using a flat, anvil-type micrometer. Exercise extreme caution to prevent damage to the critical area of the test specimen. Record and report the measured dimensions and locations of the measurements. Use the average of the multiple measurements in the stress calculation.

7.4 *Handling and Cleaning*—Exercise care in handling and storing specimens in order to avoid introducing random and severe flaws, which might occur if the specimens were allowed to impact or scratch each other. Clean the test specimens with an appropriate medium such as methanol or high-purity (>99 %) isopropyl alcohol to avoid contamination of the test environment by residual machining or processing fluids. After cleaning and drying, store the test specimens in a controlled environment such as vacuum or a dessicator in order to avoid exposure to moisture. This is necessary if testing is to be carried out in an environment other than ambient air or water. Adsorbed moisture on the test specimen surfaces can change crack growth rates.

7.5 *Number of Test Specimens*—At least ten specimens per applied stress shall be used. The total number of test specimens shall be at least 40, with at least four different applied stresses (see Section 8.3.1). The numbers of test specimens and applied stresses in this test method have been established with the intent of determining reasonable confidence limits on both time-to-failure distribution and SCG parameters.

NOTE 8—Refer to Ref (11) when a specific purpose is sought for the statistical reproducibility of SCG parameters in terms of several variables.

7.6 *Randomization of Test Specimens*—Since a somewhat large number of test specimens (a minimum of 40) with at least four different applied stresses is used in this test method, it is highly recommended that all the test specimens be randomized prior to testing in order to reduce any systematic error associated with material fabrication or specimen preparation, or both. Randomize the test specimens (using, for example, a random number generator) in groups equal to the number of applied stresses to be employed. Complete randomization may not be appropriate if the specimens stem from different billets. Trace the origin of the test specimens and use an appropriate statistical blocking scheme for distributing the specimens.

## 8. Procedure

8.1 *Test Specimen and Load Fixture Dimensions*—Choose the appropriate fixture in the specific test configurations. A fully articulating fixture is required if the specimen parallelism requirements cannot be met. Conduct 100 % inspection/measurements of the test specimens and test specimen dimensions to assure compliance with the specifications in this test

method. Measure the test specimen width,  $b$ , and depth,  $d$ . Exercise extreme caution to prevent damage to the test specimen.

8.2 Measurement of surface finish is not required, however, such information would be helpful. Methods such as contact profilometry can be used to determine the surface roughness of the test specimen faces. When quantified, report surface roughness, test conditions, and the direction of the measurement with respect to the test specimen long axis.

8.3 *Applied Stresses:*

8.3.1 *Range and Number of Applied Stress Levels:*

The choice of range and number of applied stress levels (or applied force levels) not only depends on test material, but also affects the statistical reproducibility of SCG parameters. Time to failure of advanced monolithic ceramics in constant stress testing is probabilistic. Furthermore, the scatter in time to failure is significantly greater than that in strength (11-13), typically  $(n+1)$  times the Weibull modulus of strength distribution, see Appendix X2. Hence, unlike metallic or polymeric materials, a considerable increase in the scatter of time to failure is expected for advanced monolithic ceramics, attributed to both a large strength scatter (Weibull modulus of about 10-15) and a typically high SCG parameter  $n \geq 20$ . As a consequence, testing a few test specimens at each applied stress using a few stress levels may not be sufficient to produce statistically reliable design data. On the contrary, the use of many test specimens with many applied stresses is quite time consuming or even unrealistic in some cases. In general, choose the upper limit of applied stresses that would result in corresponding time to failure  $\geq 10$  s. The choice of the lower limit of applied stresses depends on run-out times, where some of test specimens would not fail within a prescribed length of test time. The run-out time needs to be determined in the particular test program; however, experience has shown that run-out times up to 10 days are reasonable in laboratory test conditions. Choose at least four applied stresses covering at least four orders of magnitude in time. See also Appendix X3.

NOTE 9—If SCG parameters are available from constant stress-rate testing (Test Method C1368), time to failure in constant stress testing can be estimated as a function of applied stress from a prediction shown in Appendix X3. This approach, although theoretical, allows one to quickly find the range and magnitude of stresses and the run-out time to be applied. There might be some discrepancies in the prediction; however, use of this prediction can significantly reduce many uncertainties and trial-and-errors associated with selecting stresses and run-out time. If no SCG data for the test material is available, run simplified constant stress-rate testing using both high (around 10 MPa/s) and low (around 0.01 MPa/s) stress rates with at least five test specimens at each stress rate to determine fracture strengths. Then determine the corresponding SCG parameters ( $n$  and  $D_{II}$ ) based on the procedure in Test Method C1368. Use these simplified SCG data to select applied stresses and run-out time to be used in constant stress testing by following the prediction described in Appendix X3.

8.4 *Assembling Test Fixture /Specimen:*

8.4.1 Examine the bearing cylinders to make sure that they are undamaged, and that there are no reaction products (corrosion products or oxidation) that could result in uneven line loading of the test specimen or prevent the bearing cylinders from rolling. Remove and clean, or replace, the

bearing cylinders, if necessary. Avoid any undesirable dimensional changes in the bearing cylinders, for example, by inadvertently forming a small flat on the cylinder surface when abrasion (for example, abrasive paper) is used to remove the reaction products from the cylinders. The same care should be directed toward the contact surfaces in the loading and support members of the test fixture that are in contact with the bearing cylinders.

8.4.2 Carefully place each test specimen into the test fixture to avoid possible damage and contamination and to ensure alignment of the test specimen relative to the test fixture. There should be an equal amount of overhang of the test specimen beyond the outer bearing cylinders and the test specimen shall be directly centered below the axis of the applied force. Provide a way (for example, pencil marking in the test specimen or known positioning of the test specimen relative to a reference point or surface of the test fixture) to determine the fracture location of the test specimen upon fracture.

8.5 *Loading the Test Fixture/Specimen Assembly into Test Machine*—Mount the test fixture/test specimen assembly in the load train of the test machine. If necessary, slowly (~1MPa/s) apply a preload of no more than 25 % of (fast) fracture force to maintain system alignment.

8.6 *Environment*—Choose the test environment as appropriate to the test program. If the test environment is other than ambient air, supply the environmental chamber with the test medium so that the test specimen is completely exposed to the test environment. The immersion or exposure time for equilibration of the test specimen in the test environment should be determined by agreement between the parties involved in the test program. Consistent conditions (composition, supply rate, and so forth) of the test environment should be maintained throughout the test series (also refer to Section 6.3). When a corrosive liquid environment is used, put a proper protective cover onto the environment chamber (or container) to keep the test environment from splashing out of the chamber (container) upon fracture. If the tests are carried out in a humid atmosphere, the relative humidity shall not vary more than 10 % of the set humidity level during the entire test series. Determine the relative humidity in accordance with Test Method E337. Allow a sufficient period for equilibration of the test specimen in the environment. The equilibration time should be based on agreement between the parties involved in the test program and be consistent for the entire test program. This is particularly important for an environment that is chemically corrosive. When tests are conducted in ambient air, put cotton, tissues, or other appropriate material to prevent broken pieces of test specimens flying out of the test fixtures upon fracture.

8.7 *Conducting the Test*—Initiate the data acquisition. Start the test by applying a selected applied force (applied stress) with an accuracy of  $\pm 1.0$  %. Time-measuring devices, particularly when used with dead-weight test machines, should be synchronized upon the application of a test force to the test specimen. Time shall be measured at an accuracy of  $\pm 1$  % of the actual value. Record time to failure. If failure does not occur within the specific time agreed upon in the test program, record this as run-out.

8.7.1 *Recording*—Record a force versus time curve for each test in order to check the requirement of force variation of testing machines. Care should be taken in recording adequate response-rate capacity of the recorder, as described in Section 6.4.

8.8 *Post-Test Treatments*:

8.8.1 Carefully collect as many fragments as possible. Clean the fragments if necessary and store in a protective container for further analysis, including fractography.

8.8.2 *Fractography*—Fractographic analysis of fractured test specimens shall be employed to ensure that all the fracture origins are from the same population. Additional fractography may be performed to characterize the types, locations, and sizes of fracture origins as well as the flaw extensions due to slow crack growth. Follow the guidance established in Test Method C1322. See also 5.7.

## 9. Calculation

9.1 *Applied Stress*:

9.1.1 Calculate the flexural strength according to the formula for the strength of a beam in four-point  $1/4$  point flexure:

$$\sigma = \frac{3PL}{4bd^2} \quad (1)$$

where:

$\sigma$  = applied stress, MPa,

$P$  = applied force, N,

$L$  = outer (support) span, mm,

$b$  = test specimen width, mm, and

$d$  = test specimen depth, mm.

9.2 *Determining the Fatigue curve and the Slow Crack Growth Parameters  $n$  and  $D$* :

9.2.1 Use each individual time to failure, not averaged per applied stress, to determine the fatigue curve. This can be done by linear regression or maximum likelihood regression. If the data contains specimens that failed upon loading a censored analysis must be performed (left hand censoring), if the data contains run-outs, a right hand censoring must be performed. Datasets that contain both failures upon loading and run-outs must be analyzed by a two-sided censoring technique. The censoring can be performed by an iterative least squares procedure or by a maximum likelihood analysis. Several commercial statistics analysis programs and certain freeware contain censored analyses as an option (20-22).

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

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

where:

$\alpha$  = slope,

$\sigma_j$  = the  $j$ th applied stress, MPa,

$t_j$  = the  $j$ th measured time to failure,  $s$ , and

$K$  = total number of test specimens tested validly for the whole series of tests excluding the run-out test specimens.

9.2.1.2 Calculate the SCG parameter  $n$  as follows: