Designation: C1834 – 16

Standard Test Method for Determination of Slow Crack Growth Parameters of Advanced Ceramics by Constant Stress Flexural Testing (Stress Rupture) at Elevated Temperatures[1]

This standard is issued under the fixed designation C1834; 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 the slow crack growth (SCG) parameters of advanced ceramics in a given test environment at elevated temperatures in which the time-to-failure of four-point-¼ point flexural test specimens (see Fig. 1) is determined as a function of different levels of constant applied stress. This SCG constant stress test procedure is also called a slow crack growth (SCG) stress rupture test. The test method addresses the test equipment, test specimen fabrication, test stress levels and experimental procedures, data collection and analysis, and reporting requirements.

1.2 In this test method the decrease in time-to-failure with increasing levels of applied stress in specified test conditions and temperatures is measured and used to analyze the slow crack growth parameters of the ceramic. The preferred analysis 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—This test method is historically referred to in earlier technical literature as static 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 that occurs exclusively under cyclic stress loading, as defined in E1823, this test method uses the term *constant stress testing* rather than static fatigue testing.

1.3 This test method uses a 4-point-¼ point flexural test mode and 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, elastic continuous behavior, and the application of this test method to these materials is not recommended.

1.4 This test method is intended for use at elevated temperatures with various test environments such as air, vacuum, inert gas, and steam. This test method is similar to Test Method C1576 with the addition of provisions for testing at elevated temperatures to establish the effects of those temperatures on slow crack growth. The elevated temperature testing provisions are derived from Test Methods C1211 and C1465.

1.5 Creep deformation at elevated temperatures can occur in some ceramics as a competitive mechanism with slow crack growth. Those creep effects may interact and interfere with the slow crack growth effects (see 5.5). This test method is intended to be used primarily for ceramic test specimens with negligible creep. This test method imposes specific upper-bound limits on measured maximum creep strain at fracture or run-out (no more than 0.1 %, in accordance with 5.5).

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

1.7 *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

C1211 Test Method for Flexural Strength of Advanced Ceramics at Elevated Temperatures

C1239 Practice for Reporting Uniaxial Strength Data and Estimating Weibull Distribution Parameters for Advanced Ceramics

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

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

[3] 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.

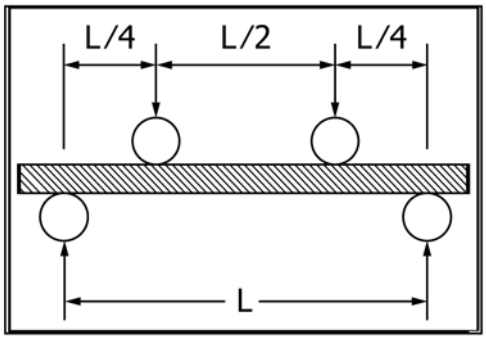

FIG. 1 Four-point-¼ Point Flexural Test Schematic

C1291 Test Method for Elevated Temperature Tensile Creep Strain, Creep Strain Rate, and Creep Time-to-Failure for Advanced Monolithic Ceramics
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 Strength 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
C1576 Test Method for Determination of Slow Crack Growth Parameters of Advanced Ceramics by Constant Stress Flexural Testing (Stress Rupture) at Ambient Temperature
E4 Practices for Force Verification of Testing Machines
E112 Test Methods for Determining Average Grain Size
E220 Test Method for Calibration of Thermocouples By Comparison Techniques
E230 Specification and Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples
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
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 Terminology C1145 and Terminology E1823 are applicable to this test method. Specific terms relevant to this test method are as follows:

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

3.1.3 *constant applied stress, σ[FL^{-2}], n*—a constant maximum flexural stress applied to a specified beam test specimen by using a constant static force with a test machine and a test fixture. **C1576**

3.1.4 *constant applied stress versus time-to-failure diagram, n*—a plot of constant applied stress against time-to-failure for experimental test data. (See Fig. 2)

3.1.4.1 *Discussion*—Constant applied stress and time-to-failure are both plotted on logarithmic scales. Data may be organized and plotted by experimental test temperature. Also called an SCG stress rupture diagram. (See Fig. 2) **C1576**

3.1.5 *constant applied stress versus time-to-failure curve, n*—a curve fitted to the values of time-to-failure at each of several applied stresses. (See Fig. 2)

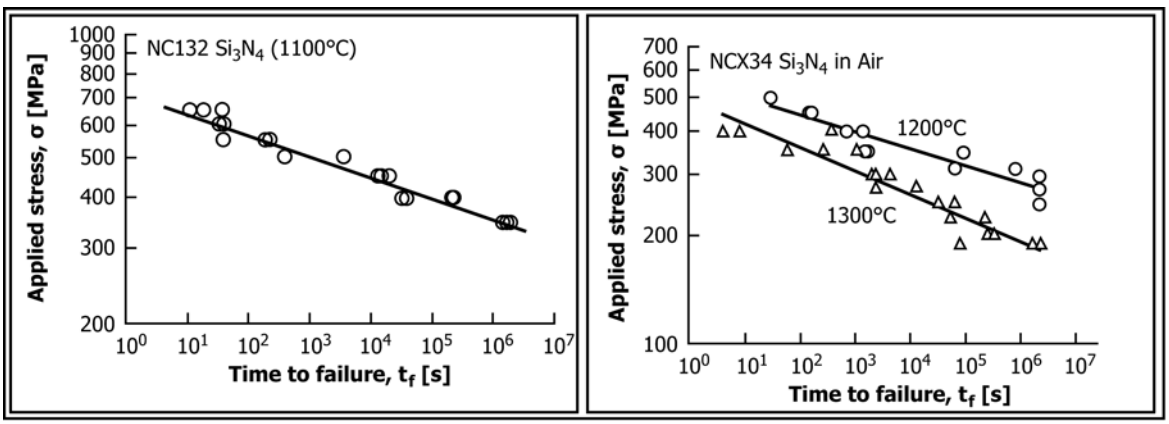

FIG. 2 Examples of Applied Stress versus Time-to-Failure Diagrams [NC132 Silicon Nitride at 1100°C in Air (Ref 28) and NCX34 Silicon Nitride at 1200°C and 1300°C in Air (Ref 29)]

3.1.5.1 *Discussion*—In the historical ceramics literature, the constant applied stress versus time-to-failure curve is often called a static fatigue curve. A more accurate descriptive name is a slow crack growth (SCG) stress rupture curve. **C1576**

3.1.6 *crack-extension resistance, K_R [$FL^{-3/2}$], G_R [FL^{-1}] or J_R [FL^{-1}], n*—a measure of the resistance of a material to crack extension expressed in terms of the stress-intensity factor, *K*; crack-extension force, *G*; or values of *J* derived using the *J*-integral concept. **E1823**

3.1.6.1 *Discussion*—The *J*-integral concept in this E1823 definition is a metal fracture concept and is not applicable to brittle ceramics.

3.1.7 *creep strain, n*—the time-dependent strain that occurs after the application of a force which is thereafter maintained constant. **C1291**

3.1.8 *dead weight test machine, n*—a mechanical testing machine which uses a load frame, lever-arms, and an adjustable weight train (with calibrated dead weights) to apply a constant known force to the test specimen over an extended period of time.

3.1.9 *flexural strength, σ_f [FL^{-2}], n*—a measure of the ultimate strength of a specified beam test specimen in flexure determined at a given stress in a particular environment. **C1576**

3.1.10 *fracture toughness, n*—a generic term for measures of resistance to extension of a crack. **E399, E1823**

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

3.1.11.1 *Discussion*—An inert condition may be obtained by testing at a low temperature, at a very fast test rate, or in an inert test environment such as vacuum, silicone oil, high purity dry N_2, or liquid nitrogen. **C1465**

3.1.12 *plane-strain fracture toughness, (critical stress intensity factor) K_{IC} [$FL^{-3/2}$], n*—the crack extension resistance under conditions of crack-tip plane strain in Mode I for slow rates of loading under predominantly linear-elastic conditions and negligible plastic-zone adjustment. **E1823**

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

Also defined as a K-R curve. **E1823**

3.1.14 *run-out, n*—a test specimen that does not fail before a prescribed test time limit. **C1576**

3.1.15 *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, C1576**

3.1.16 *slow crack growth (SCG) parameters, n*—the parameters estimated as constants in the log (*time-to-failure*) versus log (*constant applied stress*), which represent a measure of the susceptibility to slow crack growth of a material (see Appendix X1). **C1465**

3.1.17 *stress intensity factor, K_I [$FL^{-3/2}$], 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**

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

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

3.1.20 *time-to-failure, t_f [t], n*—total elapsed time from test initiation to test specimen failure/rupture for a defined test condition.

4. Significance and Use

4.1 The service life of many structural ceramic components is often limited by the subcritical growth of cracks over time, under stress at a defined temperature, and in a defined chemical environment (Refs **1-3**). When one or more cracks grow to a critical size, brittle catastrophic failure may occur in the component. Slow crack growth in ceramics is commonly accelerated at elevated temperatures. This test method provides a procedure for measuring the long term load-carrying ability and appraising the relative slow crack growth susceptibility of ceramic materials at elevated temperatures as a function of time, temperature, and environment. This test method is based on Test Method C1576 with the addition of provisions for elevated temperature testing.

4.2 This test method is also used to determine the influences of processing variables and composition on slow crack growth at elevated temperatures, as well as on strength behavior of newly developed or existing materials, thus allowing tailoring and optimizing material processing for further modification.

4.3 This test method may be used for material development, quality control, characterization, design code or model verification, time-to-failure, and limited design data generation purposes.

NOTE 2—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.

4.4 This test method and Test Method C1576 are similar and related to Test Methods C1368 and C1465; however, C1368 and C1465 use constant stress-rates (linearly increasing stress over time) to determine corresponding flexural strengths, whereas this test method and C1576 employ a constant stress (fixed stress levels over time) 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 data from 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.

4.5 The flexural stress computation in this test method is based on simple elastic beam theory, with the following assumptions: the material is isotropic and homogeneous; the moduli of elasticity in tension and compression are identical; and the material is linearly elastic. These assumptions are based on small grain size in the ceramic specimens. The grain size should be no greater than 1/50 of the beam depth as measured by the mean linear intercept method (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.

4.6 The test specimen sizes and test fixtures have been selected in accordance with Test Method C1211 which provides a balance between practical configurations and resulting errors, as discussed in Refs **4** and **5**. Test Method C1211 also specifies fixture material requirements for elevated test temperature stability and functionality.

4.7 The SCG 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 3—A variety of crack velocity functions exist in the literature. A comparison of the functions for the prediction of long-term constant stress (static fatigue) data from short-term constant stress rate (dynamic fatigue) data (Ref **6**) indicates that the exponential forms better predict the data than the power-law form. Further, the exponential form has a theoretical basis (Refs **7-10**); however, the power law form is simpler mathematically. Both forms have been shown to fit short-term test data well.

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

4.9 Slow crack growth behavior of ceramic materials can vary as a function of material properties, thermal conditions, and environmental variables. Therefore, it is essential that test results accurately reflect the effects of the 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.

4.10 Like mechanical strength, the SCG time-to-failure of advanced ceramics 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 (Refs **1, 11-13**; see Appendix X2). Hence, a proper range and number of constant applied stress levels, in conjunction with an appropriate number of test specimens, are required for statistical reproducibility and reliable design data generation (Ref **1-3**). This test method provides guidance in this regard.

4.11 The time-to-failure of a ceramic material for a given test specimen and test fixture configuration is dependent on the ceramic material's inherent resistance to fracture, the presence of flaws, the applied stress, and the temperature 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 (Refs **14, 15**), and fractography is recommended in this test method (refer to Practice C1322).

5. Interferences

5.1 Slow crack growth (SCG) may be the product of both mechanical stress and chemical driving forces. The chemical driving force for a given material may vary strongly with the chemistry and temperature of the test environment. SCG testing is conducted at temperatures and in environments representative of service conditions, so as to evaluate material performance under service 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 temperature and chemical variables of the test environment shall be controlled to minimize changes during the test. Inadequate control of temperature and environmental conditions may result in inaccurate time-to-failure data, especially for materials that are more sensitive to elevated temperatures and reactive environments.

5.2 A wide range of different interference effects can occur in slow crack growth testing at elevated temperatures (Refs **16-27**).

5.2.1 Creep damage (cavitation and micro-cracks) on or near the tensile surface of the test specimen.

5.2.2 Creep-induced non-linear stress-strain effects on the tensile surface of the test specimen.

5.2.3 Differences in creep strain on the tensile surface versus the compressive surface of the test specimen introducing non-linear stress-strain effects through the thickness of the test specimen.

5.2.4 Deviations in the linear relationship between log (constant applied stress) and log (time-to-failure) at high stress levels.

5.2.5 Oxidation induced crack healing and crack tip blunting.

5.2.6 Chemical reactions, oxidation, phase changes, and devitrification of grain boundary layers in the ceramics.

5.3 Variations in the test specimens and the experimental conditions can also act as interferences.

5.3.1 Different flaw populations between the surface and the interior of the test specimen.

5.3.2 Surface condition effects and anomalous surface flaws from specimen machining and grinding.

5.3.3 Non-uniform test specimen dimensions (dimensional variations, warp, twist, and bowing).

5.3.4 Localized fracture from contact and friction stresses at load points.

NOTE 4—These issues are discussed in detail in Annex A1 and in Test Methods C1211 and C1291.

5.4 All of these effects may change the stress conditions, the flaw populations, and the crack growth mechanisms in the test specimens. These factors need to be considered, accounted for, and controlled for each given test material and set of test conditions.

5.5 Creep deformation and effects may be a primary interference in high temperature SCG testing. Significant creep at both higher temperatures and longer test times may produce nonlinearity in stress-strain relations as well as accumulated tensile damage in flexure (Ref **11**). This, depending on the

degree of nonlinearity, may limit the applicability of linear elastic fracture mechanics (LEFM), since the resulting relationship between strength and stress derived under constant stress testing condition is based on an LEFM approach with negligible creep (maximum creep strain less than 0.1 %). Therefore creep strain should minimized as much as possible (to no more than 0.1 %), as compared to the total elastic strain at failure (see Fig. 3 and 8.9.2).

6. Apparatus

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

6.1.1 Universal test machines shall meet the system compliance requirements as cited in Annex A2 and Test Method C1211, section 6.9. Dead-weight machines do not have any compliance requirements.

6.2 *Test Fixtures*—The configurations and mechanical properties of test fixtures shall be in accordance with Test Method C1211. The materials from which the test fixtures, including bearing cylinders, are fabricated shall be effectively inert to the test environment at the test temperatures, so that they do not significantly react with or contaminate either the test specimen or the test environment. In addition, the test fixtures shall remain elastic under test temperatures and loading conditions.

NOTE 5—Various grades of silicon carbide (such as hot-pressed or sintered) and high-purity aluminas are candidate materials for test fixtures as well as load train components in the hot zone. The load-train material should also be effectively inert to the test environment. For more specific information regarding use of appropriate materials for fixtures and load train with respect to test temperatures, refer to Section 6 of Test Method C1211.

6.2.1 *Four-Point Flexure*—The four-point-¼ point fixture described in Test Method C1211, Section 6.2, shall be used in this test method (see Fig. 1). The nominal outer (support) spans (*L*) for the A, B, and C test fixtures are *L* = 20 mm, 40 mm, and 80 mm, respectively. 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 C1211 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 C1211.

6.2.3 *Semiarticulating Four-Point Fixture*—The semiarticulating four-point fixture is described in Test Method C1211. Use the semiarticulating test fixture for test specimens that meet the parallelism requirements of Test Method C1211.

6.2.4 *Fully Articulating Four-Point Fixture*—The fully articulating four-point fixture is described in Test Method C1211. Use the fully articulating test fixture for test specimens that do not meet the parallelism requirements in Test Method C1211, due to the ceramic fabrication process (as-fired, heat-treated or oxidized).

6.3 *Heating Apparatus*—The heating system (such as furnace enclosure, heating elements, thermal control, temperature measuring device, or thermocouple, or combinations thereof) shall conform to the requirements in Test Method C1211, section 6.11.

6.3.1 *Test Furnace and Temperature Readout Device*—The furnace shall be capable of maintaining the test specimen temperature within ±2°C during each testing period. The temperature readout device shall have a resolution of 1°C or smaller. The furnace system shall be such that thermal gradients are minimal along the length of the test specimen with no more than a 5°C differential from end-to-end in the test specimen.

NOTE 6—Tests are sometimes conducted in furnaces that have thermal gradients. Test specimens of smaller sizes will reduce thermal gradient problems, but it is essential to monitor the temperature along the length of the test specimen.

6.3.2 *Thermocouples:*

6.3.2.1 The specimen temperature shall be monitored by a thermocouple with its tip situated no more than 1 mm from the midpoint of the test specimen. Either a fully sheathed or exposed bead junction may be used. If a sheathed tip is used, verify that there is negligible error associated with the covering sheath.

(1) Thermocouple integrity and stability are significant concerns at elevated temperatures and long exposure times. Exposed thermocouple beads have greater sensitivity, but they may be exposed to vapors that may react with the thermocouple materials. (For example, silica vapors will react with platinum.) Beware of the use of heavy-gage thermocouple wire, thermal gradients along the thermocouple length, or

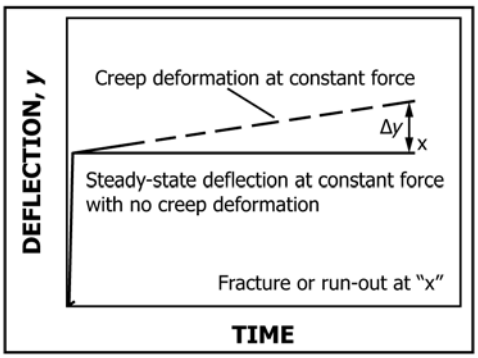

FIG. 3 Creep Deformation and Deflection at Constant Force Conditions

excessively heavy-walled insulators, all of which may lead to erroneous temperature readings.

(2) The thermocouple tip may contact the test specimen, but only if there is certainty that the thermocouple tip or sheathing material will not interact chemically with the test specimen. Thermocouples may be prone to breakage if they are in contact with the test specimen.

6.3.2.2 A separate thermocouple may be used to control the furnace, if necessary, but the test specimen temperature shall be the reported temperature of the test.

6.3.2.3 The thermocouple(s) shall be calibrated in accordance with Test Method E220 and Specification and Tables E230. The thermocouples shall be periodically checked since calibration may drift with usage or contamination.

6.3.2.4 The measurement of temperature shall be accurate to within ±5°C. The accuracy shall include the error inherent to the thermocouple as well as any errors in the measuring instruments.

NOTE 7—Resolution should not be confused with accuracy. Beware of recording instruments that read out to 1°C (resolution) but have an accuracy of only ±10°C or ±½ % of full-scale (for example, ½ % of 1200°C is 6°C).

NOTE 8—Temperature measuring instruments typically approximate the temperature-electromotive force (EMF, in millivolt) tables, and may have an error of a few degrees.

6.3.2.5 The appropriate thermocouple extension wire shall be used to connect a thermocouple to the furnace controller and temperature readout device, which shall have either a cold junction or a room-temperature compensation circuit. Special care should be directed toward connecting the extension wire with the correct polarity.

6.4 *Furnace Environmental Chamber*—The furnace may have an air, inert, vacuum, or any other gaseous environment, as required. If testing is conducted in any gaseous environment other than ambient air, an appropriate environmental chamber shall be constructed to facilitate handling, control, and monitoring of the test environment so that constant test environment 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. If the load train acts through bellows, fittings, or seals, verify that force losses or errors do not exceed 1% of the prospective failure forces.

6.5 *Deflection Measurement*—Beam deflection and outer fiber strain (strain at the outer face of the flexure beam) measurements are not needed to calculate a slow crack growth parameter. However, deflection measurements may be necessary for determining if significant creep deformation is occurring. Deflection measurement of test specimens for creep is particularly important for certain ceramic materials at higher test temperatures and longer test times and is highly recommended to ensure that maximum creep strain of those ceramic specimens is within the allowable limit (see 8.9.2).

6.5.1 Creep deformation can be measured by three methods: real-time in-situ deflection of the midpoint or load points of the test specimen, crosshead displacement, and post-test measurement of the permanent deformation of the midpoint of the test specimen.

6.5.2 Deflection-measuring equipment shall be capable of resolution and accuracy of 2×10^{-3} mm. See A2.2 for details on deflection measurement and creep strain calculation.

6.6 *Data Acquisition*—Accurate determination of the time-to-failure (or maximum test time in case of run-out) is important, since time-to-failure is the only dependent variable in this test method. Applied force versus elapsed time shall be measured and recorded during testing to ensure constant stress conditions.

6.6.1 Accurate time determination is particularly important when time-to-failure may be relatively short (<10 s). Devices to measure time-to-failure may be either digital or analog and incorporate a switching mechanism to mark the time of test specimen failure. Either analog chart recorders or digital data acquisition systems may be used for this purpose.

6.6.2 Time 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 data series. The appropriate data acquisition rate depends on the actual time-to-failure 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).

6.7 *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 inner span section, thickness/depth measurements should be made using a flat, anvil type micrometer. Ball-tipped or sharp anvil micrometers should not be used because localized surface damage (e.g., cracking) may be induced.

7. Test Specimen

7.1 *Specimen Size*—The types/configurations, dimensions, and tolerances of rectangular flexure beam specimens described in Test Method C1211 shall be used in this test method. The nominal dimensions [width (*b*), depth (*d*), and length (*l*)] for each type of test specimen are given in Table 1.

7.2 *Specimen Preparation*—Specimen fabrication and finishing methods as described in Test Method C1211, Section 7.2, shall be used in this test method. These methods are defined as application-matched machining, customary procedures, and standard procedures.

7.3 *Specimen Measurement*—It is common practice to measure the specimen dimensions post-test to prevent surface damage in the critical area (inner span section). If there is a concern about dimensional changes in test specimens from

TABLE 1 Test Specimen Dimensions (per Test Method C1211)[A,B]

Type/ Configuration	Width (*b*), mm	Depth/Thickness (*d*), mm	Length (*l*) mm, minimum
A	2.0	1.5	25
B	4.0	3.0	45
C	8.0	6.0	90

[A] Cross-sectional dimensional tolerances are ±0.05 mm for A specimens and ±0.13 mm for B and C specimens.
[B] The parallelism tolerances on the four longitudinal faces are 0.015 mm for A and B specimens and 0.03 mm for C specimens. The two end faces need not be precision machined.

oxidation/reaction layers on the surface over long test times, measure the test specimen dimensions prior to testing.

7.3.1 For measurements prior to and after testing, determine the width (b) and depth (d) at three points along the inner span of each test specimen as described in Test Method C1211, either optically or mechanically using a flat, anvil-type micrometer. Exercise extreme care in pretest measurements to prevent damage to the critical area (the inner span section) of the test specimen. Record and report the measured dimensions and locations of the measurements. Use the average of the multiple measurements (width and depth) in the stress calculation.

7.3.2 Measurement of surface finish is not required, however, such information may be helpful in assessing surface flaws. Methods such as contact profilometry may be used to determine the surface roughness of the test specimen faces. When measured, report the measured surface roughness (RMS), test method, and the direction of the measurement with respect to the long axis of the test specimen.

7.4 *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 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 a vacuum or a dessicator in order to minimize exposure to moisture. Adsorbed moisture on the test specimen surfaces may change slow crack growth rates.

7.5 *Number of Test Specimens*—The required number of test specimens depends on the desired level of statistical reproducibility of the calculated SCG parameters (n and D). The statistical reproducibility is a function of the strength scatter number (Weibull modulus), the range of applied stress levels, and the SCG parameter (n). Because of these different variables, there is no absolute rule as to the determination of the appropriate number of test specimens.

7.5.1 A minimum of ten specimens per each applied stress level is recommended in this test method with at least four different applied stress levels (4 stresses × 10 specimens = 40 specimens). The recommended number of test specimens (and applied stress levels) has been established with the intent of determining reasonable confidence limits on both time-to-failure distribution and SCG parameters. (See 8.1.1.)

NOTE 9—Refer to Ref **1** when a specific purpose is sought for the statistical reproducibility of SCG parameters.

7.6 *Randomization of Test Specimens*—Since a large number of test specimens (a recommended 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 are taken from different billets. Trace and record the source of the test specimens and use an appropriate statistical blocking scheme for distributing the specimens.

7.7 *Valid Tests*—A valid individual test is one that meets the following three requirements: *(1)* all the experimental requirements of this test method are met, *(2)* fracture occurs in the uniformly stressed section (that is, in the inner span; see 8.10.2), and *(3)* the maximum creep strain does not exceed the selected creep strain limit.

8. Procedure

8.1 *Test Preparation:*

8.1.1 *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. A minimum of ten specimens per each applied stress level is recommended in this test method with at least four different applied stress levels (4 stresses × 10 specimens = 40 specimens).

8.1.2 In general, choose an upper limit of applied stress that would result in a corresponding time-to-failure of ~10 s. The choice of the lower limit of applied stresses depends on run-out times, where some of the test specimens would not fail within a prescribed length of test time. Determine an appropriate run-out time for each particular test program, depending on the SCG mechanisms of the ceramic and the material service and temperature requirements. Reported laboratory tests of high strength, high temperature ceramics have used a range of run-out times: 10^6, 10^7, and 10^8 seconds. Choose at least four applied stresses covering at least four orders of magnitude in time.

NOTE 10—Time-to-failure of advanced monolithic ceramics in constant stress testing is probabilistic. Furthermore, the scatter in time-to-failure is significantly greater than the scatter in strengths (Refs **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 to 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 other side of the equation, the use of many test specimens with many applied stresses is quite time consuming and may be unrealistic in time and cost.

NOTE 11—If SCG parameters are available from constant stress-rate testing (Test Method C1368 and Test Method C1465), 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 may 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_d) 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.

8.1.3 For each selected stress level, calculate the necessary applied force for the dimensions of the selected test specimen and loading configuration, using the stress calculation equation (Eq 1) in 9.1.1.

8.1.4 Define a heating rate for the furnace that will minimize temperature overshoot and thermal shock to the test specimen.

8.2 *Test Specimen Inspection and Measurement*—Conduct 100 % inspection of the test specimens to assure compliance with the specifications in this test method. Specimen dimensions (width, *b*, and depth, *d*) are commonly measured post-test, to prevent pretest damage to the surfaces of the test specimen (see 7.3.1 and 8.10.2). If there is a concern about a dimensional changes in test specimens from oxidation/reaction layers on the surface over long test times, measure the test specimen dimensions prior to testing.

8.3 *Test Fixture and System Assembly:*

8.3.1 *Test Fixtures*—Choose the appropriate fixture for the specific test configurations, as described in 6.2. Use the four-point "A" fixture for the Size A specimens. Similarly, use the four-point "B" fixture for Size B specimens, and the four-point "C" fixture for Size C specimens. Use a fully articulating fixture if the specimen parallelism requirements cannot be met.

8.3.2 *Inspecting and Assembling the Test Fixture*—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 could 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, the inadvertent forming a small flat on the cylinder surface when abrasion (e.g., abrasive paper) is used to remove the reaction products from the cylinders. The same care should be directed toward the contact surfaces of the loading and support members of the test fixture that are in contact with the bearing cylinders. Assemble the test fixture, so that it is properly aligned and can articulate without restraint or significant friction.

8.3.3 *Furnace and Environmental Chamber Set Up*—Install and assemble the heating/furnace system (and environmental chamber, if used) so that it is properly aligned and functioning for test specimen heating, environmental control, and testing.

8.3.4 *Load System Set-Up*—Set up and check the load-control and force measurement devices in the test system. For dead weight systems, select and mount the required weights into the load train. For universal test machines, set the test mode to load-control.

8.4 *Test Specimen Loading and Heating:*

8.4.1 Carefully place the 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 method (e.g., 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.

8.4.2 *Loading the Test Fixture/Specimen Assembly into Test Machine*—Mount and align the test specimen/ fixture assembly in the load train of the test machine. If necessary, slowly apply a preload of no more than 25 % of the test force to maintain system alignment during deflection probe positioning and specimen heat up.

8.4.3 If test specimen deflection is to be measured (see 6.5) using a contact type of equipment, position the deflection-measurement probe(s) with its rounded tip in contact with the midpoint and/or the inner load points (tension side) of the test specimen. Exercise care to apply an appropriate contact force (see 6.5.2 and Annex A2).

8.5 An appropriate containment shield should be furnished for keeping test fragments from scattering in the furnace after fracture. If possible, retrieve the test specimens from the furnace as soon as possible after fracture in order to preserve the primary fracture surfaces for subsequent fractographic analysis.

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 atmosphere so that the test specimen is completely exposed to the test atmosphere. Consistent conditions (composition, supply rate, etc.) of the test environment should be maintained throughout the test series (see 6.4). If the tests are carried out in a humid atmosphere, the relative humidity should not vary more than 10 % (absolute) during the entire test series. At ambient temperatures, determine the relative humidity in accordance with Test Method E337. Allow a sufficient period for equilibration of the test specimen in the test environment.

8.7 *Heating to the Test Temperature*—Initiate the temperature data acquisition. Heat the test specimen to the test temperature at the selected heating rate. Temperature overshoot over the test temperature shall be strictly controlled and shall be no more than 5°C. Maintain the temperature within ± 5°C (soak time) to allow the entire system to reach thermal equilibrium. Prior to testing, the soak time should be determined experimentally at the test temperature. The soak time shall be stated in the test report.

8.8 *Hot-Furnace Loading and Heating (Optional)*—In some cases, test specimens may be loaded directly into a hot furnace, as described in section 8.4 of Test Method C1211. The fixture may be either left in the furnace for the entire time or removed partially or completely, depending on the details of the system. Exercise care to ensure that the bearing cylinders and test specimen are positioned accurately. Furthermore, exercise extreme care to ensure that possible damage associated with thermal shock shall not have any effect on strength or slow crack growth, or both, of test specimens. If needed and possible, place the deflection-measurement probe in contact with the midpoint of specimens between the two inner bearing cylinders, in accordance with 8.4.3. Determine the equilibration time of the test specimen at the test temperature experimentally prior to testing.

8.9 *Conducting the Test*—Initiate the data acquisition for force, temperature, time, and deflection (if measured). Start the test by applying the selected applied force (applied stress) with

an accuracy of ±1.0 % in a smooth, controlled manner. 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. Elapsed time shall be measured at an accuracy of ±1 % of the actual value.

8.9.1 *Recording*—Record the force and temperature versus time data for each test in order to check the requirement for constant force and temperature during the test. Care should be taken to ensure adequate response-rate capacity of the recorder, as described in 6.6. Upon specimen fracture or failure, record the time-to-failure for each test. If failure does not occur within the specified run-out time, record the elapsed time as a run-out.

8.9.2 If specimen deflection is measured during the test, record the deflection versus time data for each test.

8.9.2.1 *Deflection Increases over Time in the Deflection-Time Plot*—If the measured deflection increases over time at a constant force level (observed from the recorded deflection-time plot), creep strain/deformation is probably present (see Fig. 3). Creep strain may become dominant at higher test temperatures and longer test times. Although it is difficult to specify a general limit on maximum creep strain, it may be appropriate to limit the nominal (tensile face) creep strain to no more than 0.1 % (Ref **16** and Test Method C1465). A larger or smaller creep strain limit may be defined, based on a mutual agreement, but this shall be stated in the report. If the maximum creep strain for a given stress level is greater than the agreed upon limit, use a higher applied stress while still meeting the requirement for at least four different applied stress levels.

8.10 *Post-Test Treatments and Analysis:*

8.10.1 After fracture, carefully collect as many test specimen sections and fragments as possible. Clean the sections and fragments if necessary and store in a protective container for further analysis, including fractography and creep deflection measurement.

8.10.2 *Post-Test Specimen Dimensions*—Measure and record the width (*d*) and depth (*b*) of each test specimen to within 0.002 mm, at one point near the fracture origin and two points near the load points. In the special case where there is a concern about dimensional change of test specimens after testing due to oxidation/reaction surface layers, take the measurements prior to testing (see 7.3).

8.10.3 *Fracture Location*—Examine the location of fracture origin for each test specimen. Make certain that a valid test is one in which fracture occurs only in the uniformly stressed section (that is, the inner span).

NOTE 12—Due to the nonuniform, steep stress-gradients occurring in the sections outside the inner span, it is rarely possible to determine the exact stress level of a test specimen that fractured outside the inner span. Therefore, the test specimens that fractured outside the inner span are not recommended for use as valid data points in determining the slow crack growth parameters. In the case of multiple fractures, it is recommended to ascertain that the primary fracture occurred inside the inner span. Guidance for determining primary fracture is given in Practice C1322.

8.10.4 From a conservative standpoint, when completing a required number of test specimens at each stress level, test one replacement test specimen for each invalid test specimen (fracture outside the inner span or excessive creep deformation). If test specimens at a given stress level consistently have excessive creep strain, perform tests at a higher stress level. However, for more rigorous statistical analysis (such as Weibull statistics) with a large number of test specimens, a censoring technique may be used to deal with such anomalous data points as discussed in Practice C1239.

8.10.5 *Fractography*—Fractographic analysis of fractured test specimens may be used 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. Fractography may also show signs of creep deformation. Follow the guidance established in Test Method C1322.

8.10.6 Creep deformation can be measured from retrieved test specimens, by measuring the permanent deformation at the midpoint of the test specimen by optical or mechanical dimension measurement. (See A2.2.3.)

9. Calculation

9.1 *Applied Stress:*

9.1.1 Calculate the applied flexural stress for each test specimen according to the elastic stress formula for a beam in four-point-¼ point flexure:

$$\sigma = \frac{3PL}{4bd^2} \tag{1}$$

where:

σ = applied flexure stress, MPa,
P = applied force, N,
L = outer (support) span, mm,
b = test specimen width, mm, and
d = test specimen depth, mm.

9.1.2 *Alternate Practice*—Eq 1 neglects to compensate for thermal expansion of the fixture and specimen at elevated test temperatures, since all dimensions are taken at room-temperature. Expansion of the fixture and specimen may lead to errors of 1 to 3 % for advanced ceramic materials such as alumina, silicon carbide, silicon nitride, and zirconia. Annex A1.1 in Test Method C1211 provides a modified formula for Eq 1 and shall be used if the average thermal expansion coefficient of the fixture and the specimen are known. The use of the thermal expansion corrected equations shall be stated explicitly in the report.

9.1.3 If the test specimens edges are chamfered or rounded, and if the sizes of the chamfers or rounds exceeds the limits in 7.2.4.8 and Fig. 4 in Test Method C1161, then the strength of the beam shall be corrected in accordance with Annex A2 of Test Method C1161. The use of the chamfer corrected equations shall be stated explicitly in the report.

9.2 *Determining the Constant Applied Stress versus Time-to-Failure Curve and the Slow Crack Growth Parameters n and D:*

9.2.1 Individual time-to-failure test values for each test specimen (not the averaged time per applied stress), are used to determine the time-to-failure curve. This may be done by linear regression or maximum likelihood regression. If the data contains specimens that failed upon initial loading, a censored analysis shall be performed (left hand censoring); if the data

contains run-outs, a right hand censoring shall be performed. (See A1.3.) Datasets that contain both failures upon loading and run-outs shall be analyzed by a two-sided censoring technique. The censoring may 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 analysis option (Refs **30-32**).

9.2.2 Determination of SCG parameters depends on which crack velocity relationship is selected. The approach based on a power law relationship between crack velocity and applied stress intensity is given as the preferred method in this test method. See Appendix X1 for derivations and alternative methods.

9.2.3 Use the individual time-to-failure values t_f to determine the SCG parameters. Plot the log of the applied stress (σ in MPa) against the log of time-to-failure (t_f in s). The SCG parameters n and D_s may be determined by a linear regression analysis using all log t_f data over the complete range of individual log σ data, based on the following equation (see Appendix X1 for derivation):

$$\log t_f = -n\log\sigma + \log D_S \tag{2}$$

9.2.3.1 Include all the data points determined as valid tests in the diagram. However, do not include the run-outs or the data points in the plateau regions (see Fig. A1.1) in calculating SCG parameters. Examples of plots of log (*applied stress*) against log (*time-to-failure*) for two different ceramics at elevated temperatures are shown in Fig. 2.

NOTE 13—It seems to be more logical to plot the dependent variable, log (t_f), as a function of the independent variable, log (σ); however, it has been a long practice to plot log (σ) versus log (t_f) such as in Fig. 2. This type of diagram when determined under cyclic loading is called an *S-N* curve (E1823). This SCG test method follows this common convention in plotting data points. However, the regression shall be performed as defined in Eq 2.

NOTE 14—This test method is intended to determine only slow crack growth parameters n and D. The calculation of the parameter A (in $v = A[K_I/K_{IC}]^n$) requires knowledge of other material parameters, and is beyond the scope of this test method (see Appendix X1).

NOTE 15—This test method is primarily for test specimens with intrinsic flaws. If test specimens, however, possess any residual stresses produced by localized contact damage (e.g., particle impact or indents) or any other treatments, the estimated SCG parameters will be different and shall be denoted as such. Refer to Ref **33** for more detailed information on the analysis of slow crack growth behavior of a material containing a localized residual stress field.

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

$$\alpha = \frac{K\sum_{j=1}^{K}(\log\sigma_j \log t_j) - \left(\sum_{j=1}^{K}\log\sigma_j \sum_{j=1}^{K}\log t_j\right)}{K\sum_{j=1}^{K}(\log\sigma_j)^2 - \left(\sum_{j=1}^{K}\log\sigma_j\right)^2} \tag{3}$$

where:

α = slope of the linear regression line,
σ_j = the jth applied stress, MPa,
t_j = the jth measured time-to-failure, s, and
K = total number of test specimens tested validly for the whole series of tests excluding the plateau and run-out test specimens.

9.2.5 Calculate the SCG parameter n as follows:

$$n = -\alpha \tag{4}$$

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

$$\beta = \frac{\left(\sum_{j=1}^{K}\log t_j\right)\sum_{j=1}^{K}(\log\sigma_j)^2 - \left(\sum_{j=1}^{K}\log\sigma_j \log t_j\right)\left(\sum_{j=1}^{K}\log\sigma_j\right)}{K\sum_{j=1}^{K}(\log\sigma_j)^2 - \left(\sum_{j=1}^{K}\log\sigma_j\right)^2} \tag{5}$$

where:

β = zero intercept of the linear regression line.

9.2.7 Calculate the SCG parameter D_S as follows:

$$D_S = 10^{\beta} \tag{6}$$

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

$$SD_\alpha = \sqrt{\frac{K}{K-2}\,\frac{\sum_{j=1}^{K}(\alpha\ \log\sigma_j + \beta - \log t_j)^2}{K\sum_{j=1}^{K}(\log\sigma_j)^2 - \left(\sum_{j=1}^{K}\log\sigma_j\right)^2}} \tag{7}$$

$$SD_n = SD_\alpha \tag{8}$$

where:

SD_n = standard deviation of the SCG parameter n, and
SD_α = standard deviation of the slope, α.

9.2.9 Calculate the standard deviations of the intercept ß and of the SCG parameter D_S as follows:

$$SD_\beta = \sqrt{\frac{\sum_{j=1}^{K}(\alpha\ \log\sigma_j + \beta - \log t_j)^2 \sum_{j=1}^{K}(\log\sigma_j)^2}{(K-2)\left[K\sum_{j=1}^{K}(\log\sigma_j)^2 - \left(\sum_{j=1}^{K}\log\sigma_j\right)^2\right]}} \tag{9}$$

$$SD_{D_S} = 2.3026(SD_\beta)(10^\beta) \tag{10}$$

where:

SD_β = standard deviation of the intercept β, and
SD_{D_S} = standard deviation of the SCG parameter D_S.

9.2.10 Calculate the coefficients of variation of the SCG parameter n and of the SCG parameter D_S as follows:

$$CV_n(\%) = \frac{100(SD_n)}{n} \tag{11}$$

$$CV_{D_S}(\%) = \frac{100(SD_{D_S})}{D_S} \tag{12}$$

where:

CV_n = coefficient of variation of the SCG parameter n, and
CV_{D_S} = coefficient of variation of the SCG parameter D_S.

9.2.11 Calculate the square of the correlation coefficient (r^2) of the linear regression line as follows:

$$r^2 = \frac{\left[K\sum_{j=1}^{K}(\log\sigma_j \log t_j) - \left(\sum_{j=1}^{K}\log\sigma_j \sum_{j=1}^{K}\log t_j\right)\right]^2}{\left[K\sum_{j=1}^{K}(\log\sigma_j)^2 - \left(\sum_{j=1}^{K}\log\sigma_j\right)^2\right]\left[K\sum_{j=1}^{K}(\log t_f)^2 - \left(\sum_{j=1}^{K}\log t_f\right)^2\right]} \tag{13}$$