INTERNATIONAL

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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.

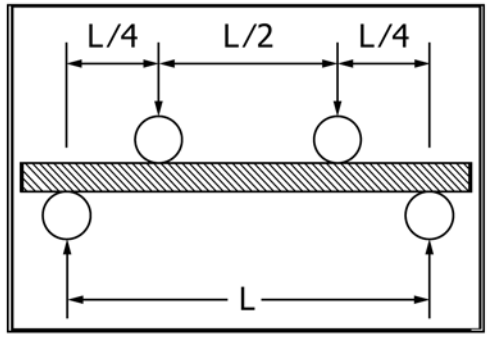

FIG. 1 Four-point-¼ Point Flexural Test Schematic

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)

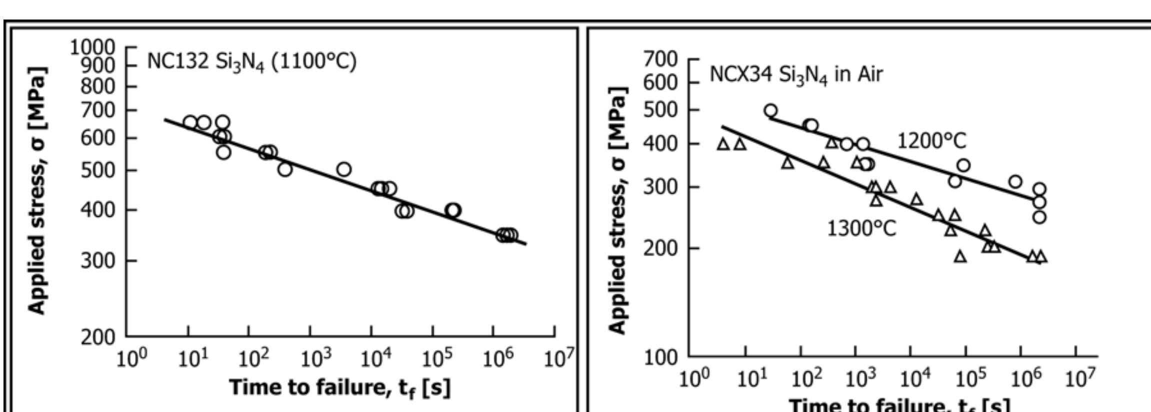

FIG. 2 Examples of Applied Stress versus Time-to-Failure Diagrams [NC132 Silicon Nitride at 1100°C in Air (Ref 28) and NCX34 Silicon Nitride at 1200°C and 1300°C in Air (Ref 29)]

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