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Standard Test Methods for Determination of Fracture Toughness of Advanced Ceramics at Ambient Temperature¹

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1. Scope

1.1 These test methods cover the fracture toughness determination of K_{Ipb} (precracked beam test specimen), K_{Isc} (surface crack in flexure), and K_{Ivb} (chevron-notched beam test specimen) of advanced ceramics at ambient temperature. The fracture toughness values are determined using beam test specimens with a sharp crack. The crack is either a straight-through crack (pb), or a semi-elliptical surface crack (sc), or it is propagated in a chevron notch (vb).*

1.1 These test methods cover the fracture toughness, K_{Ic} , determination of advanced ceramics at ambient temperature. The methods determine K_{Ipb} (precracked beam test specimen), K_{Isc} (surface crack in flexure), and K_{Ivb} (chevron-notched beam test specimen). The fracture toughness values are determined using beam test specimens with a sharp crack. The crack is either a straight-through crack formed via bridge flexure (pb), or a semi-elliptical surface crack formed via Knoop indentation (sc), or it is formed and propagated in a chevron notch (vb), as shown in Fig. 1.

NOTE 1—The terms bend(ing) and flexure are synonymous in these test methods.

1.2 These test methods determine fracture toughness values based on a force and crack length measurement (pb, sc), or a force measurement and an inferred crack length (vb). In general, the fracture toughness is determined from maximum force. Applied force and displacement or an alternative (for example, time) are recorded for the pb test specimen and vb test specimen.

1.3 These test methods are applicable to materials with either flat or with rising R-curves. The fracture toughness measured from stable crack extension may be different than that measured from unstable crack extension. This difference may be more pronounced for materials exhibiting a rising R-curve.

NOTE 2—One difference between the procedures in these test methods and test methods such as Test Method E399, which measure fracture toughness, K_{Ic} , by one set of specific operational procedures, is that Test Method E399 focuses on the start of crack extension from a fatigue precrack for metallic materials. In these test methods the test methods for advanced ceramics make use of either a sharp precrack formed via bridge flexure (pb) or via Knoop indent (sc) prior to the test, or a crack formed during the test (vb). Differences in test procedure and analysis may cause the values from each test method to be different. Therefore, fracture toughness values determined with these methods cannot be interchanged with K_{Ic} as defined in Test Method E399 and may not be interchangeable with each other.

1.4 These test methods give fracture toughness values, K_{Ipb} , K_{Isc} , and K_{Ivb} , for specific conditions of environment, test rate and temperature. The fracture toughness values, K_{Ipb} , K_{Isc} , and K_{Ivb} for a material can be functions of environment, test rate and temperature.

1.5 These test methods are intended primarily for use with advanced ceramics which are macroscopically homogeneous. Certain whisker- or particle-reinforced ceramics may also meet the macroscopic behavior assumptions.

1.6 These test methods are divided into three major parts and related sub parts as shown below. The first major part is the main body and provides general information on the test methods described, the applicability to materials comparison and qualification, and requirements and recommendations for fracture toughness testing. The second major part is composed of annexes that provide procedures, test specimen design, precracking, testing, and data analysis for each method. Annex A1 describes suggested test fixtures, Annex A2 describes the pb method, Annex A3 describes the sc method, and Annex A4 describes the vb method. The third major part consists of three appendices detailing issues related to the fractography and precracking used for the sc method.

1.2 These test methods are applicable to materials with either flat or with rising R-curves. Differences in test procedure and analysis may cause the values from each test method to be different. For many materials, such as the silicon nitride Standard Reference Material 2100, the three methods give identical results at room temperature in ambient air.

1.3 The fracture toughness values for a material can be functions of environment, test rate and temperature. These test methods give fracture toughness values for specific conditions of environment, test rate and temperature.

1.4 These test methods are intended primarily for use with advanced ceramics which are macroscopically homogeneous. Certain

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

whisker- or particle-reinforced ceramics may also meet the macroscopic behavior assumptions. Single crystals may also be tested.
 1.5 This standard begins with a main body that provides information on fracture toughness testing in general. It is followed by annexes and appendices with specific information for the particular test methods.

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1.6 Values expressed in these test methods are in accordance with the International System of Units (SI) and Practice E 880.

1.7 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

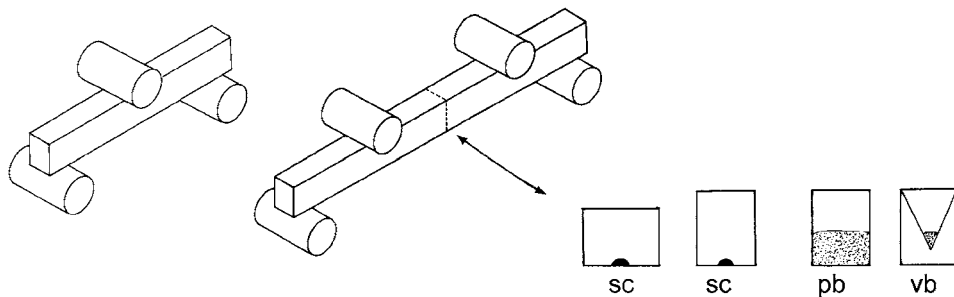
1.8 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:²

- C 1161 Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature
- C 1322 Practice for Fractography and Characterization of Fracture Origins in Advanced Ceramics
- E 4 Practices for Force Verification of Testing Machines
- E 112 Test Methods for Determining Average Grain Size
- E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

² 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.



NOTE 1—The figures on the right show the test specimen cross sections and crack types. Four-point loading may be used with all three methods. Three-point may be used with the pb and vb specimens.

FIG. 1 The Three Test Methods

- E 337 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
 E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
 E 740 Practice for Fracture Testing with Surface-Crack Tension Specimens
 E 1823 Terminology Relating to Fatigue and Fracture Testing
 IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI) (The Modern Metric System)

2.2 Reference Material:

NIST SRM 2100 Fracture Toughness of Ceramics³

3. Terminology

3.1 Definitions:

3.1.1 The terms described in Terminology E 1823 are applicable to these test methods. Appropriate sources for each definition are provided after each definition in parentheses.

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

3.1.3 *fracture toughness*—a generic term for measures of resistance of extension of a crack. (E399, E1823)

3.1.4 _____ (E 1823)

3.1.3 *R-curve*—a plot of crack-extension resistance as a function of stable crack extension.

3.1.5

3.1.4 *slow crack growth (SCG)*—sub critical crack growth (extension) which may result from, but is not restricted to, such mechanisms as environmentally-assisted stress corrosion or diffusive crack growth.

3.1.6

3.1.5 *stress-intensity factor*, $K [FL^{-3/2}]$ —the magnitude of the ideal-crack-tip stress field (stress field singularity) for a particular mode in a homogeneous, linear-elastic body. (E 1823)

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *back-face strain*—the strain as measured with a strain gage mounted longitudinally on the compressive surface of the test specimen, opposite the crack or notch mouth (often this is the top surface of the test specimen as tested)

3.2.2 *crack depth*, $a [L]$ —in surface-cracked test specimens, the normal distance from the cracked beam surface to the point of maximum penetration of crack front in the material.

3.2.3 *crack orientation*—a description of the plane and direction of a fracture in relation to a characteristic direction of the product. This identification is designated by a letter or letters indicating the plane and direction of crack extension. The letter or letters represent the direction normal to the crack plane and the direction of crack propagation.

3.2.3.1 *Discussion*—The characteristic direction may be associated with the product geometry or with the microstructural texture of the product.

3.2.3.2 *Discussion*—The fracture toughness of a material may depend on the orientation and direction of the crack in relation to the material anisotropy, if such exists. Anisotropy may depend on the principal pressing directions, if any, applied during green body forming (for example, uniaxial or isopressing, extrusion, pressure casting) or sintering (for example, uniaxial hot-pressing, hot isostatic pressing). Thermal gradients during firing can also lead to microstructural anisotropy.

3.2.3.3 *Discussion*—The crack plane is defined by letter(s) representing the direction normal to the crack plane as shown in Fig. 1, Fig. 2, and Fig. 3. The direction of crack extension is defined also by the letter(s) representing the direction parallel to the characteristic direction (axis) of the product as illustrated in Fig. 1b, Fig. 2b and Fig. 3b.

- HP = hot pressing direction (See Fig. 1)
- EX = extrusion direction (See Fig. 2)
- AXL = axial, or longitudinal axis (if HP or EX are not applicable)
- R = radial direction (See Fig. 1, Fig. 2 and Fig. 3)
- C = circumferential direction (See Fig. 1, Fig. 2 and Fig. 3)
- R/C = mixed radial and circumferential directions (See Fig. 3b)

3.2.3.4 *Discussion*—For a rectangular product, R and C may be replaced by rectilinear axes x and y , corresponding to two sides of the plate.

3.2.3.5 *Discussion*—Depending on how test specimens are sliced out of a ceramic product, the crack plane may be circumferential, radial, or a mixture of both as shown in Fig. 3.

3.2.3.6 Identification of the plane and direction of crack extension is recommended. The plane and direction of crack extension are denoted by a hyphenated code with the first letter(s) representing the direction normal to the crack plane, and the second letter(s) designating the expected direction of crack extension. See Fig. 1, Fig. 2 and Fig. 3.

3.2.3.7 *Discussion*—In many ceramics, specification of the crack plane is sufficient.

3.2.3.8 Isopressed products, amorphous ceramics, glasses and glass ceramics are often isotropic, and crack plane orientation has little effect on fracture toughness. Nevertheless, the designation of crack plane relative to product geometry is recommended. For

³ Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, <http://www.nist.gov>.

example, if the product is isopressed (either cold or hot) denote the crack plane and direction relative to the axial direction of the product. Use the same designation scheme as shown in Figs. 1 and 2, but with the letters “AXL” to denote the axial axis of the product.

3.2.3.9 If there is no primary product direction, reference axes may be arbitrarily assigned but must be clearly identified.

3.2.4 *critical crack size [L]*—in these test methods, the—The crack size at which maximum force and catastrophic fracture occur in the precracked beam (see Fig. 4) and the surface crack in flexure (see Fig. 5) configurations. In the chevron-notched test specimen (see Fig. 6) this is the crack size at which the stress intensity factor coefficient, Y^* , is at a minimum or equivalently, the crack size at which the maximum force would occur in a linear elastic, flat R-curve material.

3.2.5 3.2.4 *four-point - 1/4 point flexure*—flexure configuration where a beam test specimen is symmetrically loaded at two locations that are situated one quarter of the overall span, away from the outer two support bearings (see Fig. A1.1) (C 1161)

3.2.5 *fracture toughness K_{Ic} [$FL^{-3/2}$]*—the critical stress intensity factor, Mode I, for fracture. It is a measure of the resistance to crack extension in brittle materials.

3.2.6 *fracture toughness K_{Ipb} [$FL^{-3/2}$]*—the measured stress intensity factor corresponding to the extension resistance of a straight-through crack formed via bridge flexure of a sawn notch or Vickers or Knoop indentation(s). The measurement is performed according to the operational procedure herein and satisfies all the validity requirements. (See Annex A2).

3.2.7 *fracture toughness K_{Isc} or K_{Isc}^* [$FL^{-3/2}$]*—the measured (K_{Isc}) or apparent (K_{Isc}^*) stress intensity factor corresponding to the extension resistance of a semi-elliptical crack formed via Knoop indentation, for which the residual stress field due to indentation has been removed. The measurement is performed according to the operational procedure herein and satisfies all the validity requirements. (See Annex A3).

3.2.8 *fracture toughness K_{Ivb} [$FL^{-3/2}$]*—the measured stress intensity factor corresponding to the extension resistance of a stably-extending crack in a chevron-notched test specimen. The measurement is performed according to the operational procedure herein and satisfies all the validity requirements. (See Annex A4).

3.2.9 *minimum stress-intensity factor coefficient, Y_{min}^** —the minimum value of Y^* determined from Y^* as a function of dimensionless crack length, $\alpha = a/W$.

3.2.10 *pop-in*—in these test methods, the—The sudden formation or extension of a crack without catastrophic fracture of the test specimen, apparent from a force drop in the applied force-displacement curve. Pop-in may be accompanied by an audible sound or other acoustic energy emission.

3.2.11 *precrack*—a crack that is intentionally introduced into the test specimen prior to testing the test specimen to fracture.

3.2.12 *small crack*—a crack is defined as being small when all physical dimensions (in particular, with length and depth of a surface crack) are small in comparison to a relevant microstructural scale, continuum mechanics scale, or physical size scale. The specific physical dimensions that define “small” vary with the particular material, geometric configuration, and loadings of interest.

(E1823)

3.2.13 *stable crack extension*—controllable, time-independent, noncritical crack propagation.

3.2.13.1

3.2.12.1 *Discussion*—The mode of crack extension (stable or unstable) depends on the compliance of the test specimen and test fixture; the test specimen and crack geometries; R-curve behavior of the material; and susceptibility of the material to slow crack growth.

3.2.14

3.2.13 *three-point flexure*—flexure configuration where a beam test specimen is loaded at a location midway between two support bearings (see Fig. A1.2) (C 1161)

3.2.15

3.2.14 *unstable crack extension*—uncontrollable, time-independent, critical crack propagation.

3.3 *Symbols:*

3.3.1 a —as used in these test methods, crack depth, crack length, crack size.—crack depth, crack length, crack size.

3.3.2 a_o —as used in these test methods, chevron tip dimension, vb method, Fig. 6 and—chevron tip dimension, vb method, Fig. A4.1.

3.3.3 a_1 —as used in these test methods, chevron dimension, vb method, Fig. 6, ($a_{\text{chevron dimension, vb method, } (a_1 = (a_{a_1} + a_{a_2})/2)$), Fig. A4.1

3.3.4 a_{11} —as used in these test methods, chevron dimension, vb method, Fig. 6 and—chevron dimension, vb method, Fig. A4.1.

3.3.5 a_{12} —as used in these test methods, chevron dimension, vb method, Fig. 6 and—chevron dimension, vb method, Fig. A4.1.

3.3.6 $a_{0.25}$ —as used in these test methods, crack length measured at 0.25B, pb method, Fig. 4—crack length measured at 0.25B, pb method, Fig. A4.2.

3.3.7 $a_{0.50}$ —as used in these test methods, crack length measured at 0.5B, pb method, Fig. 4—crack length measured at 0.5B, pb method, Fig. A4.2.

3.3.8 $a_{0.75}$ —as used in these test methods, crack length measured at 0.75B, pb method, Fig. 4—crack length measured at 0.75B, pb method, Fig. A4.2.

- 3.3.9 a/W —normalized crack size.
- 3.3.10 B —~~as used in these test methods, the~~ the side to side dimension of the test specimen perpendicular to the crack length (depth) as shown in Fig. A2.4, Fig. 5A3.7, and Fig. 6A4.1.
- 3.3.11 c —~~as used in these test methods, crack half width, sc method, see Fig. 5 and Fig. A3.2~~ crack half width, sc method, Fig. A3.7.
- 3.3.12 d —~~as used in these test methods, length~~ length of long diagonal for a Knoop indent, length of a diagonal for a Vickers indent, sc method.
- 3.3.13 E —elastic modulus.
- 3.3.14 $f(a/W)$ —function of the ratio a/W , pb method, four-point flexure, Eq A2.6.
- 3.3.15 F —indent force, sc method.
- 3.3.16 F_c —chamfer correction factor, sc method
- 3.3.17 $g(a/W)$ —function of the ratio a/W , pb method, three-point flexure, Eq A2.2 and Eq A2.4.
- 3.3.17h—~~as used in this standard, depth of Knoop or Vickers indent, sc method, Eq A3.1.~~
- 3.3.18 h —depth of Knoop or Vickers indent, sc method, Eq A3.1.
- 3.3.19 $H_1(a/c, a/W)$ —a polynomial in the stress intensity factor coefficient, for the precrack periphery where it intersects the test specimen surface, sc method, Eq A3.7.
- 3.3.19
- 3.3.20 $H_2(a/c, a/W)$ —a polynomial in the stress intensity factor coefficient, for the deepest part of a surface crack, sc method, see Eq A3.5.
- 3.3.20
- 3.3.21 K_I —stress intensity factor, Mode I.
- 3.3.21
- 3.3.22 K_{Ic} —fracture toughness, critical stress intensity factor, Mode I.
- 3.3.23 K_{Ipb} —fracture toughness, pb method, Eq A2.1 and Eq A2.3.
- 3.3.22
- 3.3.24 K_{Isc} —fracture toughness, sc method, Eq A3.9.
- 3.3.23
- 3.3.25 K_{Ivb} —fracture toughness, vb method, Eq A4.1.
- 3.3.24
- 3.3.26 L —test specimen length, Figs. A2.1 and A3.1 Fig. A2.1 and Fig. A3.1.
- 3.3.25L1
- 3.3.27 L_1, L_2 —precracking fixture dimensions, pb method, Fig. A2.2.
- 3.3.26
- 3.3.28 $M(a/c, a/W)$ —a polynomial in the stress intensity factor coefficient, sc method, see Eq A3.4.
- 3.3.27
- 3.3.29 P —force.
- 3.3.28
- 3.3.30 P_{max} —force maximum.
- 3.3.29
- 3.3.31 $Q(a/c)$ —a polynomial function of the surface crack ellipticity, sc method, Eq A3.3.
- 3.3.30
- 3.3.32 $S(a/c, a/W)$ —factor in the stress intensity factor coefficient, sc method, Eq A3.8.
- 3.3.31
- 3.3.33 S_o —outer span, three- or four-point test fixture. Figs. A1.1 and A1.2.
- 3.3.32
- 3.3.34 S_i —inner span, four-point test fixture, Fig. A1.1.
- 3.3.33
- 3.3.35 t —notch thickness, pb and vb method.
- 3.3.34—~~notch thickness, pb and vb method, Fig. A2.3 and Fig. A4.1.~~
- 3.3.36 W —the top to bottom dimension of the test specimen parallel to the crack length (depth) as shown in Fig. 4A2.4, Fig. 5A3.7, and Fig. 6A4.1.
- 3.3.35
- 3.3.37 Y —stress intensity factor coefficient.
- 3.3.36
- 3.3.38 Y^* —stress intensity factor coefficient for vb method.
- 3.3.37
- 3.3.39 Y_{max} —maximum stress intensity factor coefficient occurring around the periphery of an assumed semi-elliptical precrack, sc method

3.3.38

3.3.40 Y_{min}^* —minimum stress intensity factor coefficient, vb method, Eq A4.2-A4.5

3.3.39

3.3.41 Y_d —stress intensity factor coefficient at the deepest part of a surface crack, sc method, Eq A3.2

3.3.40

3.3.42 Y_s —stress intensity factor coefficient at the intersection of the surface crack with the test specimen surface, sc method, Eq A3.6

4. Summary of Test Methods

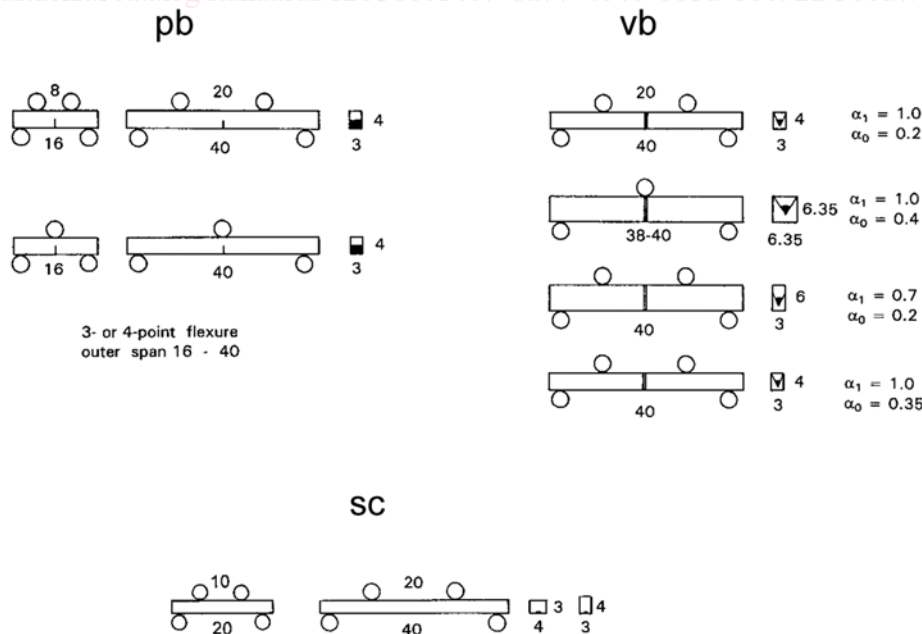
4.1 These methods involve application of force to a beam test specimen in three- or four-point flexure. The test specimen either contains a sharp crack initially or develops one during loading. The equations for calculating the fracture toughness have been established on the basis of elastic stress analyses of the test specimen configurations described for each test method.

4.2 *Precracked Beam Method*—A straight-through precrack is created in a beam test specimen via the bridge-flexure technique. In this technique the precrack is extended from median cracks associated with one or more Vickers indents or a shallow sawed notch. The fracture force of the precracked test specimen as a function of displacement or alternative (for example, time, back-face strain, or actuator displacement) in three- or four-point flexure is recorded for analysis. The fracture toughness, K_{Ipb} , is calculated from the fracture force, the test specimen size and the measured precrack size. Background information concerning the basis for development of this test method may be found in Refs. (1) and (2).

4.1 These methods involve application of force to a beam test specimen in three- or four-point flexure. The test specimen is very similar to a common flexural strength test specimen. The test specimen either contains a sharp crack initially (pb, sc) or develops one during loading (vb). The equations for calculating the fracture toughness have been established on the basis of elastic stress analyses of the test specimen configurations. Specific sizes are given for the test specimens and the flexure fixtures. Some are shown in Fig. 2. Annex A2-Annex A4 have more specific information and requirements for each method.

4.2 Each method has advantages and disadvantages that are listed in the following three paragraphs. These factors may be considered when choosing a test method. Nuances and important details for each method are covered in the specific annexes. Experience with a method increases the chances of obtaining successful outcomes. Some trial and error may be necessary with a new material or the first time a method is used, so it is wise to prepare extra test specimens. Background information concerning the basis for development of these test methods may be found in Refs. (1-6).

4.3 *Surface Crack in Flexure Method*—A beam test specimen is indented with a Knoop indenter and polished (or hand ground), while maintaining surface parallelism, until the indent and associated residual stress field are removed. The fracture force of the test specimen is determined in four-point flexure and the fracture toughness, K_{Isc} , is calculated from the fracture force, the test specimen size, and the measured precrack size. Background information concerning the basis for development of this test method may be found in Refs. (3) and (4).



NOTE 1—Other three-point and four-point spans are permitted for the sc and pb methods.

FIG. 2 Primary Test Specimen and Fixture Configurations: General Schematic

the bridge-flexure technique. In this technique the precrack is extended from median cracks associated with one or more Vickers or Knoop indentations or a shallow saw notch. The fracture force of the precracked test specimen as a function of displacement or alternative (for example, time, back-face strain, or actuator displacement) in three- or four-point flexure is recorded for analysis. The fracture toughness, K_{Ipb} , is calculated from the fracture force, the test specimen size and the measured precrack size. Advantages of this method are that it uses a classic fracture configuration and the precracks are large and not too difficult to measure. A disadvantage is that a special bridge precracking fixture is required to pop in a precrack. A well designed and well crafted bridge precracking fixture is needed to obtain good precracks. Another disadvantage is that large compression loads are needed to pop in the precrack. Another minor disadvantage is that once precracked, the test specimen must be handled with care since only a small force is necessary to break it. The precrack size must be measured. This is not difficult for most ceramics, but dye penetration techniques may be needed for some materials (e.g., those with coarse grain microstructures) if the precrack does not stand out clearly.

4.4 *Surface Crack in Flexure Method*—A beam test specimen is indented with a Knoop indenter and polished (or hand ground), until the indent and associated residual stress field are removed. The fracture force to break the test specimen is determined in four-point flexure and the fracture toughness, K_{Isc} , is calculated from the fracture force, the test specimen size, and the measured precrack size. An advantage of this method is that the precracks are very small and may not be much larger than the natural strength limiting flaws in the material, so the measured fracture toughness is appropriate for the size scale of the natural flaws. A disadvantage of this method is that fractographic techniques are required to measure the small precracks and some skill and fractographic equipment is needed. Another disadvantage is that this method will not work on very soft or porous ceramics since precracks will not form beneath the indenter that is used to pop in a precrack. The method also will not work in materials whose rough microstructure prevents the measurement of the precrack.

4.5 *Chevron-Notched Beam Method*—A chevron-notched beam is loaded in either three- or four-point flexure. Applied force versus displacement or an alternative (for example, time, back-face strain, or actuator displacement) is recorded in order to detect unstable fracture, since the test is invalid for unstable conditions. The fracture toughness, K_{Ivb} , is calculated from the maximum force applied to the test specimen after extension of the crack in a stable manner. Background information concerning the basis for the development of this test method may be found in Refs. (5) and (6). K_{Ivb} is calculated from the maximum force applied to the test specimen after extension of the crack in a stable manner. The crack forms during the loading sequence. One major advantage of this method is that it is not necessary to measure the crack size. On the other hand, it is essential that stable crack extension be obtained during the test. This may be difficult for some ceramics with large elastic moduli and small fracture toughness values. The chevron notch must be machined very carefully as described in this method in order to facilitate stable crack extension and also to satisfy the requirements for a valid test result. A stiff machine/load train/fixture is often necessary to obtain stable crack extension.

NOTE 3—The K_{Isc} —The fracture toughness of many ceramics varies as a function of the crack extension occurring up to the relevant maximum force. The actual crack extension to achieve the minimum stress intensity factor coefficient (Y^*_{min}) of the chevron notch configurations described in this method is 0.68 to 0.93 mm. This is likely to result in a fracture toughness value in the upper region of the R-curve.

<https://standards.iteh.ai/catalog/standards/sist/30ee14c7-8a77-4c40-883a-0be92f936ed7/astm-c1421-09>

5. Significance and Use

5.1 These test methods may be used for material development, material comparison, quality assessment, and characterization.

5.2 The pb and the vb fracture toughness values provide information on the fracture resistance of advanced ceramics containing large sharp cracks, while the sc fracture toughness value provides this information for small cracks comparable in size to natural fracture sources.

NOTE 4—Cracks of different sizes may be used for the sc method. If the fracture toughness values vary as a function of the surface crack size it can be expected that

5.1 Fracture toughness, K_{Ic} , is a measure of the resistance to crack extension in a brittle material. These test methods may be used for material development, material comparison, quality assessment, and characterization.

5.2 The pb and the vb fracture toughness values provide information on the fracture resistance of advanced ceramics containing large sharp cracks, while the sc fracture toughness value provides this information for small cracks comparable in size to natural fracture sources. Cracks of different sizes may be used for the sc method. If the fracture toughness values vary as a function of the crack size it can be expected that K_{Isc} will differ from K_{Ipb} and K_{Ivb} .

6. Interferences

6.1 *R-curve*—The microstructural features of advanced ceramics can cause rising R-curve behavior. For such materials the three test methods are expected to result in different fracture toughness values. These differences are due to the amount of crack extension prior to the relevant maximum test force, P_{max} , (see 9.8), or they are due to the details of the precracking methods. For materials tested to date the fracture toughness values generally increase in the following order: K_{Isc} , K_{Ipb} , K_{Ivb} (7). However, there is insufficient experience to extend this statement to all materials. In the analysis of the vb method it is assumed that the material has a flat (no) R-curve. If significant R-curve behavior is suspected, then the sc method should be used for estimates of small-crack fracture toughness, whereas the vb test may be used for estimates of longer-crack fracture toughness. The pb fracture toughness may reflect either short- or long-crack length fracture toughness depending on the precracking conditions. For materials with a flat

(no) R-curve the values of K_{Ipb} , K_{Isc} , and K_{Ivb} are expected to be similar. are expected to be the same. NIST Standard Reference Material 2100 has a flat R-curve and $K_{Ipb} = K_{Isc} = K_{Ivb}$.

6.2 *Time-Dependent Phenomenon and Environmental Effects*—The values of K_{Ipb} , K_{Isc} , K_{Ivb} , for any material can be functions of test rate because of the effects of temperature or environment. Static forces applied for long durations can cause crack extension at K_I values less than those measured in these methods. The rate of, and level at which, such crack extension occurs can be changed by the presence of an aggressive environment, which is material specific. This time-dependent phenomenon is known as slow crack growth (SCG) in the ceramics community. SCG can be meaningful even for the relatively short times involved during testing and can lead to measured fracture toughness values less than the inherent resistance in the absence of environmental effects. This effect may be significant even at ambient conditions and can often be minimized or emphasized by selecting a fast or slow test rate, respectively, or by changing the environment. The recommended testing rates specified are an attempt to limit environmental effects.

6.3 *Stability*—The stiffness of the test set-up can affect the fracture toughness value. This standard permits measurements of fracture toughness under either unstable (sc, pb) or stable (sc, pb, vb) conditions. Stiff testing systems will promote stable crack extension. A stably-extending crack may give somewhat lower fracture toughness values (8,9). —This standard permits measurements of fracture toughness whereby the crack propagates unstably (sc and pb methods) or stably (sc, pb, vb). The stiffness of the test set-up can affect whether the crack grows stably or unstably. There is limited data that suggests a stably propagating crack may give a slightly lower fracture toughness value than an unstably propagating crack (1-3).

Processing details, service history, and environment may alter the fracture toughness of the material.

6.4 Processing details, service history, and environment may alter the fracture toughness of the material.

7. Apparatus

7.1 *Testing*—Test the test specimens in —Use a testing machine that has provisions for autographic recording of force applied to the test specimen versus either test specimen load or centerline deflection or time. The force accuracy of the testing machine shall be in accordance with Practice E 4.

7.2 *Deflection Measurement*—When —Deflection measurements are optional, but if determined, measure test specimen deflection for the pb and vb close to the crack. The deflection gauge should be capable of resolving 1×10^{-3} mm (1 μ m) while exerting a contacting force of less than 1 % of the maximum test force, P_{max} .

NOTE 5—If 3—If actuator displacement (stroke) is used to infer deflection of the test specimen for the purposes of assessing stability, caution is advised. Actuator displacement (stroke), although sometimes successfully used for this purpose (9), may not be as sensitive to changes of fracture behavior in the test specimen as measurements taken on the test specimen itself, such as back-face strain, load-point displacement, or displacement at the crack plane (10).

7.3 *Recording Equipment*—Provide a means for automatically recording the applied force-displacement or load-time test record, (such as a X-Y recorder). For digital data acquisition sampling rates of 500 Hz or greater are recommended.

7.4 *Fixtures*—Use four-point or three-point test fixtures to force the pb and vb test specimens. Use four-point test fixtures only to force the sc test specimens. In addition, use a precracking fixture for the pb method. —The pb and vb test specimens may be tested in either three-point or four-point fixtures. Annex A2 and Annex A3 give the recommended span sizes for these two methods, respectively. sc test specimens shall only be tested in four-point fixtures. Bend fixtures designed for flexural strength testing in accordance with Test Method C 1161 are suitable, but this test method allows spans and configurations not in C 1161. A bridge precracking fixture is also necessary for the pb method. It is described in Annex A2.

NOTE 6—Hereafter in this document the term four-point flexure will refer to the specific case of 1/4-(that is, quarter) point flexure.

7.4.1 The schematic of a four-point test fixture is shown in

7.4.1 The four-point test fixture (see Fig. A1.1, as specified in Test Method C1161 where the recommended outer and inner spans are) for the pb, vb, or sc methods shall conform to the general fixture requirements of Test Method C 1161. The recommended outer and inner spans are $S_o = 40$ mm and $S_i = 20$ mm, respectively. The minimum outer and inner spans shall be ≥ 20 mm, respectively, but this standard allows other span sizes provided that the minimum outer and inner spans shall be $S_o = 20$ mm and $S_i = 10$ mm, respectively. The outer rollers shall be free to roll outwards and the inner rollers shall be free to roll inwards. The rolling movement minimizes frictional restraint effects which can cause flexure errors of 3 to 20%. Place the rollers initially against their stops and hold them in position by low-tension springs (such as rubber bands). Roller pins shall have a hardness of 40 Rockwell C or greater. Other fixtures are acceptable, however, roller pins shall be free to roll and meet the criteria specified in 7.4.2. ≥ 10 mm, respectively. The outer rollers shall be free to roll outwards and the inner rollers shall be free to roll inwards. Place the rollers initially against their stops and hold them in position by low-tension springs or rubber bands or magnets. Roller pins shall have a hardness of 40 Rockwell C or greater.

7.4.2 The length of each roller shall be at least three times the test specimen dimension, B . The roller diameter shall be 4.5 ± 0.5 mm. The rollers shall be parallel to each other within 0.015 mm over either the length of the roller or a length of $3B$ or greater.

7.4.3 If the test specimen parallelism requirements set forth in Fig. A2.1 and Fig. A3.1 are not met, use an alternate fully-articulating fixture.

7.4.4 The fixture shall be capable of maintaining the test specimen alignment to the tolerances specified in 9.6 are not met, use a fully-articulating fixture as described in C 1161.

7.4.5A suggested three-point test fixture design is shown in

7.4.4 The fixture shall be capable of maintaining the test specimen alignment to the tolerances specified in Annex A2-Annex A4.

7.4.5 A three-point test fixture (see Fig. A1.2. Choose the outer support span, S_o , such that $16 \leq S_o \leq 40$ mm. Since $W = 4$ mm (the top to bottom dimension of the test specimen parallel to the crack length), then the fixture although ≤ 10 . For the vb method, W can range from 4 mm to 6.35 mm depending on the specimen type in Annex A4. Choose an outer span, S_o , should not be less than 16 mm. For limits of validity of S_o , refer to the appropriate appendix. The outer two rollers shall be free to roll outwards to minimize friction effects. The middle flexure roller shall be fixed. Alternatively, a rounded knife edge with diameter in accordance with, such that $4 \leq \frac{S_o}{W} \leq 10$. The outer two rollers shall be free to roll outwards to minimize friction effects. The middle flexure roller shall be fixed. Alternatively, a rounded knife edge with diameter in accordance with 7.4.2 may be used in place of the middle roller.

NOTE7—If stable crack extension is desired in the pb test, then displacement control mode and a stiff test system and load train may be required. The specific stiffness requirements are dependent on the test specimen dimensions, elastic modulus (E) and the precrack length (see A2.1.1.2 and Refs. (8) and (9).) A test system compliance of less than or equal to 3.3×10^{-8} m/N (including load cell and fixtures) may be required for a typical stable pb test. (See Refs. (8) and (9).)

NOTE8—A stiff test system with displacement control and a stiff load train may be required to obtain stable crack extension for the vb test (Fig. A4.3b or Fig. A4.3c). Without such stable crack extension the test is invalid (Fig. A4.3a). See also A4.3.6. A test system compliance of less than or equal to 4.43×10^{-5} m/N (including load cell and fixtures) is adequate for most vb tests. Stable crack extension is essential for a valid vb test. A test system compliance of less than or equal to 4.43×10^{-5} m/N (including load cell and fixtures) is adequate for most vb tests. Stable crack extension is not required for the pb test, but if it is desired, then a stiff load train may be required. See Refs. (8) and (9).

7.5 *Dimension-Measuring Devices*—Micrometers and other devices used for measuring test specimen dimensions shall be accurate and precise to 0.0025 mm or better. Flat, anvil-type micrometers with resolutions of 0.0025 or less shall be used for test specimen dimensions. Ball-tipped or sharp-anvil micrometers are not recommended as they may damage the test specimen surface by inducing localized cracking. Non-contacting (for example, optical comparator, light microscopy, etc.) measurements are recommended for crack, pre-crack or notch measurements, or all of these.

7.6 A conventional hardness testing machine is needed for the sc method in order to make an indentation-induced precrack. A conventional hardness machine may also be used for making a starter flaw for pb test specimens.

7.7 A bridge precracking fixture is needed for precracking pb specimens. See Annex A2.

8. Test Specimen Configurations, Dimensions and Preparation

8.1 *Test Specimen Configuration*—Three precrack configurations are equally acceptable: a straight-through pb-crack, a semi-elliptical sc-crack, or a vb-chevron notch. These configurations are shown in Fig. 4, Fig. 5, and Fig. 6. Details of the crack geometry are given in the Annexes (Test Specimens—Three precrack configurations are equally acceptable: a straight-through pb-crack, a semi-elliptical sc-crack, or a vb-chevron notch. These configurations are shown in Figs. 1 and 2. Details of the crack geometry, the specimen dimensions, and preparation requirements are given in Annex A2 for the pb, Annex A3 for the sc, and Annex A4 for the vb)

8.2 *Test Specimen Dimensions*—Specific dimensions, tolerances and finishes along with additional test specimen geometries for each method are detailed in the appropriate annex for the vb.

NOTE9—A typical “plastic” (or deformation) zone, if such exists, is no greater than a fraction of a micrometre in most ceramics, thus the specified sizes are large enough to meet generally-accepted plane strain requirements at the crack tip (see Test Method E399).

8.3 *Test Specimen Preparation*—Machining aspects unique to each test method are contained in the appropriate annex. 6—A typical “plastic” (or deformation) zone, if such exists, is no greater than a fraction of a micrometer in most ceramics, thus the specified sizes are large enough to meet generally-accepted plane strain requirements at the crack tip from a plasticity viewpoint.

9. General Procedures—General Procedures for Test Methods and Calculations

9.1 *Number of Tests*—Complete a minimum of four valid tests for each material and testing condition. Complete a minimum of five valid tests for each material and testing condition. It is prudent to prepare more than 5 test pieces. This will provide specimens for practice tests to determine the best precracking conditions and also provide specimens to make up for unsuccessful or invalid tests. More specimens are needed if environment, testing rate, or precrack sizes will be varied.

9.2 *Valid Tests*—A valid individual test is one which meets all the following requirements: all the general testing requirements of this standard as listed in—A valid individual test is one which meets all the general testing requirements in 9.2.1, and all the specific testing requirements for a valid test of the particular test method as specified in the appropriate annex.

9.2.1 A valid test shall meet the following general requirements in addition to the specific requirements of the particular test (A2.6, A3.6 or A4.6): A valid test shall meet the following general requirements.

9.2.1.1 Test machine shall have provisions for autographic recording of force versus deflection or time, and the test machine shall have an accuracy in accordance with Practice E 4 (7.1).

9.2.1.2 Test fixtures shall comply with specifications of 7.4.

9.2.1.3 Dimension-measuring devices shall comply with specifications of 7.5.

9.2.1.4 Test specimens shall be aligned to comply with 9.6.

9.2.1.5 Test rate shall be in conformance with 9.7.

9.3 *Environmental Effects*—If susceptibility to environmental degradation, such as slow crack growth, is a concern, tests should be performed and reported at two different test rates, or in appropriately different environments

NOTE 10—If used, the two test rates should differ by two to three orders of magnitude (or greater). Alternatively, choose different environments such that the expected effect is small in one case (for example, inert dry nitrogen) and large in the other case (that is, water vapor). If an effect of the environment is detected, select the fracture toughness values measured at the greater test rates or in the inert environment. —If susceptibility to environmental degradation, such as slow crack growth, is a concern, tests should be performed and reported at two different test rates, or in appropriately different environments. Testing in an inert environment (dry nitrogen, argon, or vacuum) can eliminate environmental effects. Susceptibility to slow crack growth can be assessed by testing at two different testing rates in an air or water environment. The rates should differ by two to three orders of magnitude (or greater), however, attainment of stable crack extension in vb may be difficult at high rates. Alternatively, the susceptibility can be assessed by choosing different environments such that the expected effect is small in one case (for example, inert dry nitrogen) and large in the other case (that is, water vapor). If an effect of the environment is detected, select the fracture toughness values measured at the greater test rates or in the inert environment. An example of the effect of environment on the fracture toughness of alumina is given in Refs (10) and (31).

9.4 *R-curve*—When rising R-curve behavior is to be documented, two different test methods with different amounts of stable crack extension should be used.

NOTE 11—The pb and sc tests typically have less stable crack extension than the vb test.

9.5 *Test Specimen Measurements*—Measure and report all applicable test specimen dimensions to 0.002 mm. For a valid test the dimensions shall conform to the tolerances shown in the applicable figures and to the requirements in the specific annexes.

9.6 *Test Specimen Alignment*—Place the test specimen in the three- or four-point flexure fixture. Align the test specimen so that it is centered directly below the axis of the force application.

9.6.1 *Three-point Flexure*—pb and vb methods: The plane of the crack shall be centered under the middle roller within 0.5 mm. Measure the span within 0.5% of S_0 . Align the center of the middle roller so that its line of action shall pass midway between the two outer rollers within 0.1 mm. Seat the displacement indicator close to the crack plane. Alternatively, use actuator (or crosshead) displacement, back-face strain, or a time sweep.

NOTE 12—For short spans (for example, $S_0=16$ mm) and $S_0/W=4.0$ in three-point flexure using the pb method, errors of up to 3% in determining the critical mode I stress intensity factor may occur because of misalignment of the middle roller, misalignment of the support span or angularity of the precrack at the extremes of the tolerances allowed in 9.6.1 (11, 12). —When rising R-curve behavior is to be documented, two different test methods with different amounts of stable crack extension should be used and the results compared. The pb and sc tests typically have less stable crack extension than the vb test.

9.5 *Test Specimens and Fracture Experiments*— Specific test specimen measurements, procedures, and calculations are in Annex A2-Annex A4.

9.6.2 *Four-Point Flexure - pb, sc, and vb Methods*—The plane of the crack shall be located within 1.0 mm of the midpoint between the two inner rollers, S_i . Measure the inner and outer spans to within 0.1 mm. Align the midpoint of the two inner rollers relative to the midpoint of the two outer rollers to within 0.1 mm. For the pb and vb methods, seat the displacement indicator close to the crack plane. Alternatively, use actuator (or crosshead) displacement (stroke), back-face strain or a time sweep.

9.7

9.6 *Test Rate*—Test the test specimen so that one of the test rates determined in 9.3 will result in a rate of increase in stress intensity factor between 0.1 and 2.75 MPa $\sqrt{m/s}$. Applied force, or displacement (actuator or stroke) rates, or both, corresponding to these stress intensity factor rates are discussed in the appropriate annex. Other test rates are permitted if environmental effects are suspected in accordance with 9.3.

9.8 *Force Measurement*—Measure the relevant maximum test force, P_{\max} .

9.8.1 For the pb and sc test methods, the relevant maximum force is the greatest force occurring during the test.

9.8.2 For the vb test method, the relevant maximum force is measured as the maximum force occurring during the stable crack extension (See Fig. A4.3b and c). Ignore the maximum force due to a pop-in or crack jump. (See Fig. A4.3b). In some cases the relevant maximum force may not be the greatest force occurring during the test.

9.9 *Humidity*

9.7 *Humidity and Temperature*—Measure the temperature and humidity according to Test Method E 337.

9.10 *Test Specimen Examination*—On completion of the test, separate the test specimen halves and inspect the fracture surfaces for out-of-plane fracture, crack shape irregularities or any other imperfection that may have influenced the test result.

9.11 *Dimension Measurement*—Measure the crack or precrack dimensions of the pb or sc test specimen after fracture as specified in the appropriate annex.

10. Report

10.1 For each test specimen report the following information:

10.1.1 Test specimen identification,

- 10.1.2 Form of product tested, and materials processing information, if available,
- 10.1.3 Mean grain size, if available, by Test Method E 112 or other appropriate method,
- ~~10.1.4 Environment of test, relative humidity, temperature, and crack plane orientation;~~
- 10.1.4 Environment of test, relative humidity, temperature,
- 10.1.5 Test specimen dimensions: B and W ,
- 10.1.5.1 For the pb test specimen crack length, a , and notch thickness, t , if applicable,
- 10.1.5.2 For the sc test specimen the crack dimensions a and $2c$,
- 10.1.5.3 For the vb test specimen the notch parameters, a_0 and a_{11} and a_{12} and the notch thickness, t ,
- 10.1.6 Test fixture specifics,
- 10.1.6.1 Whether the test was in three- or four-point flexure,
- 10.1.6.2 Outer span, S_o , and inner span (if applicable), S_i ,
- 10.1.7 Applied force or displacement rate,
- 10.1.8 Measured inclination of the crack plane as specified in the appropriate annex,
- 10.1.9 Relevant maximum test force, P_{max} , as specified in the appropriate annex,
- 10.1.10 Testing diagrams (for example, applied force vs. displacement) as required,
- 10.1.11 Number of test specimens tested and the number of valid tests,
- ~~10.1.12 Fracture toughness value with statement of validity;~~
- 10.1.12 Fracture toughness values for each valid test with a statement confirming that all tests were indeed valid,
- 10.1.13 Additional information as required in the appropriate annex, and
- 10.2 Mean and standard deviation of the fracture toughness for each test method used.
- ~~10.3 Reporting Templates—Suggested reporting templates for conveniently listing pertinent data and results for the three different test methods are shown in Fig. 7, Fig. 8, and Fig. 9.~~
- 10.3 Crack plane and direction of crack propagation as appropriate (see Appendix X5).

11. Precision and Bias

11.1 *Precision*—The precision of a fracture toughness measurement is a function of the precision of the various measurements of linear dimensions of the test specimen and test fixtures, and the precision of the force measurement. The within-laboratory (repeatability) and between-laboratory (reproducibility) precisions of some of the fracture toughness procedures in this test method have been determined from inter-laboratory test programs (13, 14). For specific dependencies of each test method, refer to the appropriate annex. More information about the precisions of the three test methods are in the Annexes A2 – A4.

11.2 *Bias*—Standard Reference Material (SRM) 2100 from the National Institute of Standards and Technology may be used to check for laboratory test result bias. The laboratory average value may be compared to the certified reference value of fracture toughness. SRM 2100 is a set of silicon nitride beam test specimens for which the mean fracture toughness is 4.57 MPa√m and is certified to within 2.3% at a 95% confidence level. The last line of Table 2 in this standard includes some results obtained on SRM 2100 test specimens. Additional data (not shown) confirms that virtually identical results are obtained with the three test methods in this standard when used on SRM 2100. As discussed in—Standard Reference Material (SRM) 2100 from the National Institute of Standards and Technology may be used to check for laboratory test result bias. The laboratory average value may be

TABLE 1 Fracture Toughness Values of Sintered Silicon Carbide (Hexoloy SA) in MPa √m

(n) = Number of test specimens tested
 \pm = 1 Standard Deviation
 ? = quantity unknown

Precracked Beam (pb)	Surface Crack in Flexure (sc)	Chevron-Notch (vb)	Ref
2.54 ± 0.20 (3)	2.69 ± 0.08 (6) ^A	2.62 ± 0.06 (6) (A config.) 2.68 ± 0.03 (a2) (B config.)	^{A,B} using II-UW material, vintage 1985
2.58 ± 0.08 (4)	2.76 ± 0.08 (4) ^A	2.61 ± 0.05 (6) (A config.) 2.46 ± 0.03 (5) (C config.)	^{A,B} using JAS material, vintage 1980
...	3.01 ± 0.35 (3) ^C	2.91 ± 0.31 (3) (B config.)	^D

^AG. D. Quinn and J. A. Salem, "Effect of Lateral Cracks Upon Fracture Toughness Determined by the Surface crack in Flexure Method," *J. Am. Ceram. Soc.*, in 85 [4] pressp. 873 – 880, July 20042.

^BJ. A. Salem, L. J. Ghosn, M. G. Jenkins, and G. D. Quinn, "Stress Intensity Factor Coefficients for Chevron-Notched Flexure Specimens," *Ceramic Engineering and Science Proceedings*, 20 [3] 1999, pp. 503–512.

^CThis data set may have been susceptible to overestimation of the sc fracture toughness due to the interference of vestigial lateral cracks.

^DA. Ghosn, M. G. Jenkins, K. W. White, A. S. Kobayashi, and R. C. Bradt, "Elevated-Temperature Fracture Resistance of a Sintered α -Silicon Carbide," *J. Am. Ceram. Soc.*, 72 [2] pp. 242–247, 1989.

compared to the certified reference value of fracture toughness of $4.57 \text{ MPa}\sqrt{\text{m}} \pm 0.11 \text{ MPa}\sqrt{\text{m}}$ (or 2.3 %) at a 95 % confidence level. SRM 2100 is a set of five silicon nitride beam test specimens. Identical results are obtained with the three test methods in this standard when used with SRM 2100.

11.3 *Variation in Results with Test Method for Other Materials*—As discussed in 1.4, 6.1 and 6.2, for some materials K_{Ipb} , K_{Isc} and K_{Ivb} values may differ from each other (for example, (15)). Nevertheless, a comparison of test results obtained by the three different methods is instructive. Such comparisons are shown in Tables 1 and 2. The experimental procedures used in the studies cited in Tables 1 and 2 varied somewhat and were not always in accordance with this standard, although the data are presented here for illustrative purposes. Table 1 contains results for sintered silicon carbide, an advanced ceramic which is known to be insensitive to environmental effects in ambient laboratory conditions. This material is also known to have a fracture toughness independent of crack size (flat R-curve). Table 2 contains results for a hot-pressed silicon nitride which has little or no dependence of fracture toughness on crack size and which also usually had negligible sensitivity to environmental effects in ambient laboratory conditions. The hot-pressed silicon nitride results are notably consistent. Some of the variability is due to differences in fracture toughness between billets of this material (See footnotes 1 and 2 in Table 2). The results of the last line in Table 2 were generated from a single billet identified as “C.”-curve).

12. Keywords

12.1 advanced ceramics; chevron notch; fracture toughness; precracked beam; surface crack in flexure

ANNEXES

(Mandatory Information)

A1. SUGGESTED TEST FIXTURE SCHEMATICS

A1.1 See Fig. A1.1 and Fig. A1.2.

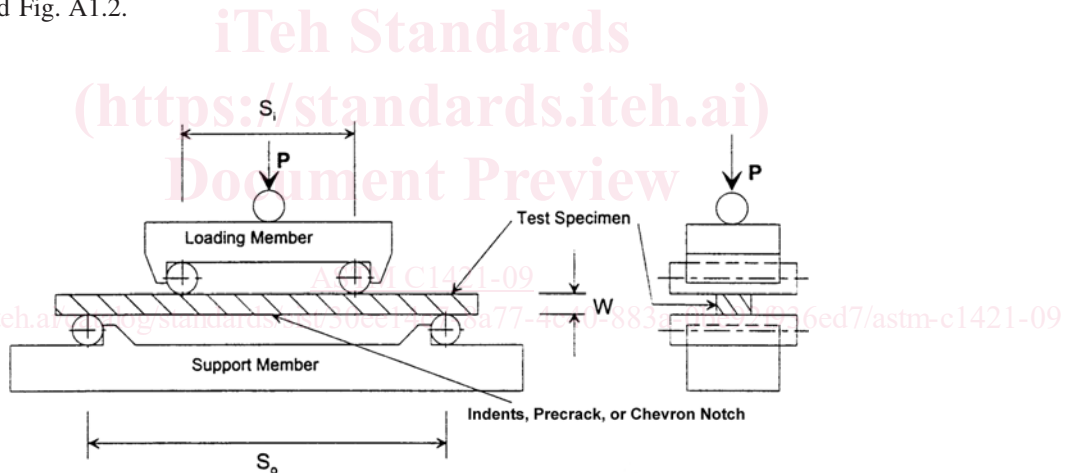


FIG. A1.1 Four-point test fixture schematic which illustrates the general requirements for a semi-articulating fixture.

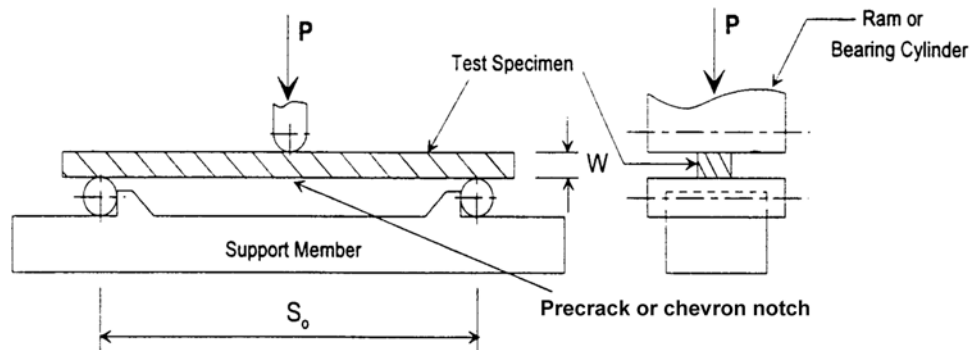


FIG. A1.2 Three-point test fixture schematic which illustrates the general requirements of the test fixture.