# Standard Test Methods for Determination of Fracture Toughness of Advanced Ceramics at Ambient Temperature<sup>1</sup>

This standard is issued under the fixed designation C 1421; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon  $(\epsilon)$  indicates an editorial change since the last revision or reapproval.

# 1. Scope

1.1 These test methods cover the fracture toughness determination of  $K_{Ipb}$ (precracked beam specimen),  $K_{Isc}$  (surface crack in flexure), and  $K_{Ivb}$ (chevron-notched beam specimen) of advanced ceramics at ambient temperature. The fracture toughness values are determined using beam 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).

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 load and crack length measurement (pb, sc), or a load measurement and an inferred crack length (vb). In general, the fracture toughness is determined from maximum load. Load and displacement or an alternative (for example, time) are recorded for the pb specimen and vb 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 E 399, which measure fracture toughness,  $K_{\rm Ic}$ , by one set of specific operational procedures, is that Test Method E 399 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 loading (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  $K_{\rm Ic}$  as defined in Test Method E 399 and may not be interchangeable with each other.

1.4 These test methods give fracture toughness values,  $K_{\rm Ipb}$ ,  $K_{\rm Isc}$ , and  $K_{\rm Ivb}$ , for specific conditions of environment, test rate and temperature. The fracture toughness values,  $K_{\rm Ipb}$ ,  $K_{\rm Isc}$ , and

 $K_{\mathrm{Ivb}}$  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, 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.

Main Body Scope	Section 1
Referenced Documents	2
Terminology (including definitions, orientation and symbols)	3
Summary of Test Methods Significance and Use 12d53e7db1d/astm-c142	1-49
Interferences	6
Apparatus	7
Specimen Configurations, Dimensions and Preparations	8
General Procedures	9
Report (including reporting tables)	10
Precision and Bias	11
Annexes	
Test Fixture Geometries	A1
Special Requirements for Precracked Beam Method	A2
Special Requirements for Surface Crack in Flexure Method	A3
Special Requirements for Chevron Notch Flexure Method	A4
Appendices	
Precrack Characterization, Surface Crack in Flexure Method	X1
Complications in Interpreting Surface Crack in Flexure Precracks	X2
Alternative Precracking Procedure, Surface Crack in Flexure Method	Х3

- 1.7 Values expressed in these test methods are in accordance with the International System of Units (SI) and Practice E 380.
- 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.

<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee C-28 on Advanced Ceramics and is the direct responsibility of Subcommittee C28.01 on Properties and Performance.

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#### 2. Referenced Documents

- 2.1 ASTM Standards:
- C 1161 Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature<sup>2</sup>
- C 1322 Practice for Fractography and Characterization of Fracture Origins in Advanced Ceramics<sup>2</sup>
- E 4 Practices for Load Verification of Testing Machines<sup>3</sup>
- E 112 Test Methods for Determining Average Grain Size<sup>3</sup>
- E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods<sup>4</sup>
- E 337 Test Method for Measured Humidity with a Psychrometer<sup>5</sup>
- E 380 Practice for Use of International System of Units (SI) (the Modernized Metric System)<sup>3</sup>
- E 399 Test Method for Plane-Strain Fracture Toughness of Metallic Materials<sup>3</sup>
- E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method<sup>4</sup>
- E 740 Practice for Fracture Testing with Surface-Crack Tension Specimens<sup>3</sup>
- E 1823 Terminology Relating to Fracture Testing<sup>3</sup>
- 2.2 Military Standards and Handbooks
- MIL-HDBK-790 Fractography and Characterization of Fracture Origins in Advanced Structural Ceramics<sup>6</sup>

# 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.
- 3.1.3 *fracture toughness*—a generic term for measures of resistance of extension of a crack. (E 399, E 1823)
- 3.1.4 *R-curve*—a plot of crack-extension resistance as a function of stable crack extension.
- 3.1.5 *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 stress-intensity factor, K [FL<sup>3/2</sup>]—the magnitude of the ideal-crack-tip stress field (stress field singularity) for a particular mode in a homogeneous, linear-elastic body.
  - 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 specimen, opposite the crack or notch mouth (often this is the top surface of the specimen as tested)

- 3.2.2 *crack depth, a* [*L*]—in surface-cracked 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)

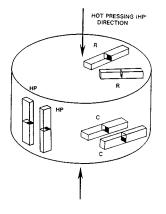
AXL = axial, or longitudinal axis (if HP or EX are not applicable)

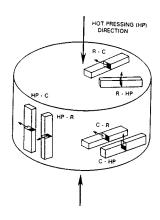
R = radial direction (See Fig. 1, Fig. 2 and Fig. 3)

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 specimens are sliced out of a ceramic product, the crack plane may be





<sup>&</sup>lt;sup>2</sup> Annual Book of ASTM Standards, Vol 15.01.

<sup>&</sup>lt;sup>3</sup> Annual Book of ASTM Standards, Vol 03.01.

<sup>&</sup>lt;sup>4</sup> Annual Book of ASTM Standards, Vol 14.02.

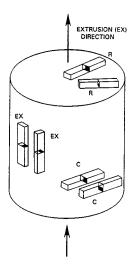
<sup>&</sup>lt;sup>5</sup> Annual Book of ASTM Standards, Vol 07.01. 11.03, and 15.09.

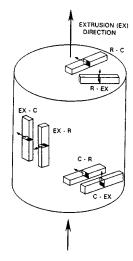
<sup>&</sup>lt;sup>6</sup> Available from Standardization Documents, Order Desk, Bldg. 4, Section D, 7000 Robbins Ave., Philadelphia, PA 19111-5094.

a) Crack plane designated, only b) Crack plane and direction of crack extension designated

Note 1—Precracked beam specimens are shown as examples. The small arrows denote the direction of crack growth.

FIG. 1 Crack Plane Orientation Code for Hot-Pressed Products





a) Crack plane designated, only

Note 1-Precracked beam specimens are shown as examples. The small arrows denote the direction of crack growth.

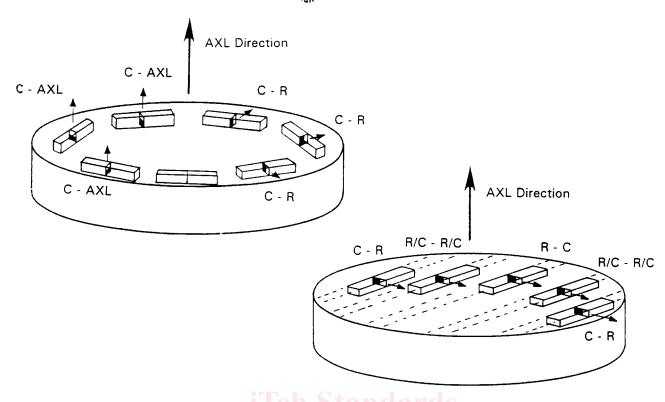
#### FIG. 2 Crack Plane Orientation Code for Extruded Products

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 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 crack size at which maximum load 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 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 load would occur in a linear elastic, flat R-curve material.
- 3.2.5 four-point 1/4 point flexure—loading configuration where a beam 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)
- 3.2.6 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 loading 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<sup>-3/2</sup>]—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 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 b) Crack plane and direction of crack extension designated dimensionless crack length,  $\alpha = a/W$ .
  - 3.2.10 pop-in—in these test methods, the sudden formation or extension of a crack without catastrophic fracture of the test specimen, apparent from a load drop in the load-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 induced into the test specimen prior to testing the 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. (E 1823)
  - 3.2.13 stable crack extension—controllable, timeindependent, noncritical crack propagation.
  - 3.2.13.1 *Discussion*—The mode of crack extension (stable or unstable) depends on the compliance of the specimen and test fixture; the specimen and crack geometries; R-curve behavior of the material; and susceptibility of the material to slow crack growth.
  - 3.2.14 three-point flexure—loading configuration where a beam specimen is loaded at a location midway between two support bearings (see Fig. A1.2) (C 1161)
  - 3.2.15 unstable crack extension—uncontrollable, timeindependent, critical crack propagation.
    - 3.3 Symbols:
  - 3.3.1 a—as used in these test methods, 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 Fig. A4.1.
  - 3.3.3  $a_1$ —as used in these test methods, chevron dimension, vb method, Fig. 6,  $(a_1 = (a_{11} + a_{12})/2)$ .
  - 3.3.4  $a_{11}$ —as used in these test methods, chevron dimension, vb method, Fig. 6 and Fig. A4.1.
  - 3.3.5  $a_{12}$ —as used in these test methods, chevron dimension, vb method, Fig. 6 and Fig. A4.1.
    - 3.3.6  $a_{0.25}$ —as used in these test methods, crack length



- a) Specimens cut circumferentially All crack planes are "C," but direction of crack extension is either radial, "R" or axial, "AXL"
- b) Specimens prepared from parallel slices.
   Crack planes and direction of crack extension are "R" or "C" or mixed depending on the location

Note 1—The R/C mix shown in b) is a consequence of the parallel slicing of the specimens from the product.

Note 2—Precracked beam specimens are shown as examples. The small arrows denote the direction of crack growth.

FIG. 3 Code for Crack Plane and Direction of Crack Extension in Specimens with Axial Primary Product Direction

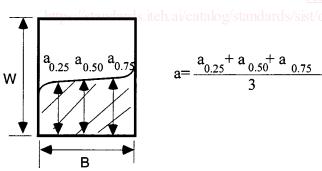


FIG. 4 Cross Section of a pb Specimen Showing the Precrack Configuration ( $a_{0.25}$ ,  $a_{0.50}$ ,  $a_{0.75}$  are the Points for Crack Length Measurements)

measured at 0.25B, pb method, Fig. 4.

- 3.3.7  $a_{0.50}$ —as used in these test methods, crack length measured at 0.5B, pb method, Fig. 4.
- 3.3.8  $a_{0.75}$ —as used in these test methods, crack length measured at 0.75B, pb method, Fig. 4.
  - 3.3.9 *a/W*—normalized crack size.
- 3.3.10 *B*—as used in these test methods, the side to side dimension of the specimen perpendicular to the crack length (depth) as shown in Fig. 4, Fig. 5, and Fig. 6.
- $3.3.11\ c$ —as used in these test methods, crack half width, sc method, see Fig. 5 and Fig. A3.2.

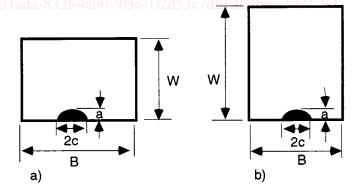


FIG. 5 a and b Cross Section of sc Specimens Showing the Precrack Configurations for Two Orientations

- 3.3.12 *d*—as used in these test methods, 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 loading, Eq A2.6.
  - 3.3.15 F—indent load, sc method.
- 3.3.16 g(a/W)—function of the ratio a/W, pb method, three-point loading, Eq A2.2 and Eq A2.4.

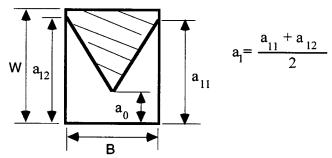


FIG. 6 Cross Section of a vb Specimen Showing the Notch Configuration

- 3.3.17 h—as used in this standard, depth of Knoop or Vickers indent, sc method, Eq A3.1.
- 3.3.18  $H_1(a/c, a/W)$ —a polynomial in the stress intensity factor coefficient, for the precrack periphery where it intersects the specimen surface, sc method, Eq A3.7.
- 3.3.19  $H_2(a/c, a/W)$ —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  $K_{\rm I}$ —stress intensity factor, Mode I.
- 3.3.21  $K_{Ipb}$ —fracture toughness, pb method, Eq A2.1 and Eq A2.3.
  - 3.3.22  $K_{Isc}$ —fracture toughness, sc method, Eq A3.9.
  - 3.3.23  $K_{Ivb}$ —fracture toughness, vb method, Eq A4.1.
  - 3.3.24 *L*—specimen length, Figs. A2.1 and A3.1.
- 3.3.25 *L*1, *L*2—precracking fixture dimensions, pb method, Fig. A2.2.
- 3.3.26 M(a/c, a/W)—a polynomial in the stress intensity factor coefficient, sc method, see Eq A3.4.
  - 3.3.27 *P*—load.
  - $3.3.28 P_{\text{max}}$ —load maximum.
- 3.3.29 Q(a/c)—a polynomial function of the surface crack ellipticity, sc method, Eq A3.3.
- 3.3.30 S(a/c, a/W)—factor in the stress intensity factor coefficient, sc method, Eq A3.8.
- $3.3.31~S_{\rm o}$ —outer span, three- or four-point flexure fixture. Figs. A1.1 and A1.2.
  - 3.3.32 S<sub>i</sub>—inner span, four-point flexure fixture, Fig. A1.1.
  - 3.3.33 *t*—notch thickness, pb and vb method.
- 3.3.34 *W*—the top to bottom dimension of the specimen parallel to the crack length (depth) as shown in Fig. 4, Fig. 5, and Fig. 6.
  - 3.3.35 Y—stress intensity factor coefficient.
  - 3.3.36 *Y*\*—stress intensity factor coefficient for vb method.
- 3.3.37  $Y_{\rm max}$ —maximum stress intensity factor coefficient occurring around the periphery of an assumed semi-elliptical precrack, sc method
- 3.3.38  $Y^*_{min}$ —minimum stress intensity factor coefficient, vb method, Eq A4.2-A4.5
- 3.3.39  $Y_d$ —stress intensity factor coefficient at the deepest part of a surface crack, sc method, Eq A3.2
- 3.3.40  $Y_s$ —stress intensity factor coefficient at the intersection of the surface crack with the specimen surface, sc method, Eq A3.6

### 4. Summary of Test Methods

4.1 These methods involve application of load to a beam specimen in three- or four-point flexure. The 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 specimen configurations described for each test method.

- 4.2 Precracked Beam Method—A straight-through precrack is created in a beam specimen via the bridge-loading 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 load of the precracked 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,  $K_{\text{Ipb}}$ , is calculated from the fracture load, the specimen size and the measured precrack size. Background information concerning the basis for development of this test method may be found in Refs. (1)<sup>7</sup> and (2).
- 4.3 Surface Crack in Flexure Method—A beam 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 load of the specimen is determined in four-point flexure and the fracture toughness,  $K_{\rm Isc}$ , is calculated from the fracture load, the 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).
- 4.4 Chevron-Notched Beam Method—A chevron-notched beam is loaded in either three- or four-point flexure. Load 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,  $K_{\text{Ivb}}$ , is calculated from the maximum load applied to the 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).

Note 3—The fracture toughness of many ceramics varies as a function of the crack extension occurring up to the relevant maximum load. 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.

# 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  $K_{Isc}$  will differ from  $K_{Ipb}$  and  $K_{Ivb}$ .

 $<sup>^{7}\,\</sup>mathrm{The}$  boldface numbers given in parentheses refer to a list of references at the end of the text.

#### 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 differing fracture toughness values. These differences are due to the amount of crack extension prior to the relevant maximum test load,  $P_{\text{max}}$ , (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:  $K_{Isc}$ ,  $K_{Inb}$ ,  $K_{Ivb}$  (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
- 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 loads applied for long durations can cause crack extension at K<sub>I</sub> 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 which may be significant even at ambient conditions 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).
- 6.4 Processing details, service history, and environment may alter the fracture toughness of the material.

### 7. Apparatus

- 7.1 Loading—Load specimens in a testing machine that has provisions for autographic recording of load applied to the specimen versus either specimen load line or centerline deflection or time. The accuracy of the testing machine shall be in accordance with Practice E 4.
- 7.2 Deflection Measurement—When determined, measure specimen deflection for the pb and vb close to the crack. The deflection gauge should be capable of resolving  $1\times10^{-3}$  mm (1  $\mu$ m) while exerting a contacting force of less than 1 % of the maximum test load,  $P_{\rm max}$ .

- Note 5—If actuator displacement (stroke) is used to infer deflection of the specimen for the purposes of assessing stability, caution is advised. Actuator displacement (stroke), although sometimes successfully used for this purpose (9), generally may not be as sensitive to changes of fracture behavior in the specimen as measurements taken on the 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 load-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 flexure fixtures to load the pb and vb specimens. Use four-point flexure fixtures only to load the sc specimens. In addition, use a precracking fixture for the pb method.

Note 6—Hereafter in this document the term four-point flexure will refer to the specific case of ½-(i.e., quarter) point loading.

- 7.4.1 The schematic of a four-point flexure fixture is shown in Fig. A1.1, as specified in Test Method C 1161 where the recommended outer (support) and inner (loading) spans are  $S_o = 40$  mm and  $S_i = 20$  mm, respectively. The minimum outer (support) and inner (loading) spans shall be  $S_o = 20$  mm and  $S_i = 10$  mm, respectively. The outer (support) rollers shall be free to roll outwards and the inner (loading) rollers shall be free to roll inwards. The rolling movement minimizes frictional restraint effects which can cause loading 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.
- 7.4.2 The length of each roller shall be at least three times the specimen dimension, B. The roller diameter shall be  $4.5\pm0.5$  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 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 specimen alignment to the tolerances specified in 9.6.
- 7.4.5 A suggested three-point flexure fixture design is shown in Fig. A1.2. Choose the outer support span,  $S_o$ , such that  $4 \le \frac{S_o}{W} \le 10$ , although  $S_o$  should not be less than 16 mm. For limits of validity of  $S_o$ , refer to the appropriate appendix. The outer two support rollers shall be free to roll outwards to minimize friction effects. The middle loading 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.
- 7.5 Compliance of Test Machine and Loading Arrangement: 7.5.1 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 specimen dimensions, elastic modulus (E) and the precrack length (see A2.1.1.2 and Refs. (8) and (9).)

Note 7—A test system compliance of less than or equal to  $3 \times 10^{-8}$  m/N (including load cell and fixtures) may be required for a typical stable

pb test. (See Refs. (8) and (9).)

7.5.2 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.

Note 8—A test system compliance of less than or equal to  $4.4\times10^{-5}$  m/N (including load cell and fixtures) is adequate for most chevron beam tests.

7.6 Dimension-Measuring Devices—Micrometers and other devices used for measuring specimen dimensions shall be accurate and precise to the level required in the appropriate annex. Flat, anvil-type micrometers may be used for specimen dimensions. Ball-tipped or sharp-anvil micrometers are not recommended as they may damage the 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.

# 8. Specimen Configurations, Dimensions and Preparation

- 8.1 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 (Annex A2 for the pb, Annex A3 for the sc, and Annex A4 for the vb)
- 8.2 Specimen Dimensions—Specific dimensions, tolerances and finishes along with additional specimen geometries for each method are detailed in the appropriate annex.

Note 9—A typical "plastic" (or deformation) zone, if such exists, is no greater than a fraction of a millimeter in most ceramics, thus the specified sizes are large enough to meet generally-accepted plane strain requirements at the crack tip (see Test Method E 399).

8.3 Specimen Preparation—Machining aspects unique to each test method are contained in the appropriate annex.

### 9. General Procedures

- 9.1 *Number of Tests*—Complete a minimum of four valid tests for each material and testing condition.
- 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 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):
- 9.2.1.1 Test machine shall have provisions for autographic recording of load versus deflection or time, and the test machine shall have an accuracy in accordance with Practice E 4 (7.1).
- 9.2.1.2 Load fixtures (7.4) shall have inner and outer rollers free to roll as required in 7.4.1 and 7.4.5, have roller pins with a hardness of 40 Rockwell C or greater (7.4.1), have rollers that have lengths at least three times the specimen dimension, B, diameters of 4.5  $\pm$  0.5 mm, with each roller parallel to each other within 0.015 mm over either the length of the roller or a length of 3B or greater (7.4.2), be capable of maintaining the

specimen alignment to the tolerances specified in 9.6 (7.4.4).

- 9.2.1.3 Dimension-measuring devices (7.6) shall be accurate and precise to the level required in the appropriate annex with all applicable dimensions measured and reported.
- 9.2.1.4 Specimen shall be aligned (9.6) such that the plane of the crack shall be centered under the center roller within 3 % of  $S_o$  for three-point loading of pb and vb specimens (9.6.1) and shall be located within 1.0 mm of the midpoint between the two inner rollers,  $S_i$  for four-point loading of pb, sc and vb specimens (9.6.2).
- 9.2.1.5 Test rate shall be (9.3, 9.7) such that one of the test rates shall result in a rate of increase in stress intensity factor between 0.1 and 2.75 MPa  $\sqrt{m}$ /s.
- 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 (e.g., 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.

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 Specimen Measurements—Measure and report all applicable 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 *Specimen Alignment*—Place the specimen in the threeor four-point fixture. Align the specimen so that it is centered directly below the axis of the load application.
- 9.6.1 Three-point Loading—pb and vb methods: The plane of the crack shall be centered under the center roller within 3 % of  $S_o$ . Measure the span within 0.5 % of  $S_o$ . Align the center of the (loading) roller so that its line of action shall pass midway between the two outer (support) 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 loading 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 center roller, misalignment of the support span or angularity of the precrack at the extremes of the tolerances allowed in 9.6.1 (11, 12).

9.6.2 Four-Point Loading - 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 (loading) rollers relative to the midpoint of the two outer (support) 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 Test Rate—Test the 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. Load, 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 Load Measurement—Measure the relevant maximum test load,  $P_{\rm max}$ .
- 9.8.1 For the pb and sc test methods, the relevant maximum load is the greatest load occurring during the test.
- 9.8.2 For the vb test method, the relevant maximum load is measured as the maximum load occurring during the stable crack extension (See Fig. A4.3b and c). Ignore the maximum load due to a pop-in or crack jump. (See Fig. A4.3b). In some cases the relevant maximum load may not be the greatest load occurring during the test.
- 9.9 *Humidity*—Measure the temperature and humidity according to Test Method E 337.
- 9.10 Specimen Examination—On completion of the test, separate the 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 specimen after fracture as specified in the appropriate annex.

# 10. Report

- 10.1 For each specimen report the following information:
- 10.1.1 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,
  - 10.1.5 Specimen dimensions: B and W,
- 10.1.5.1 For the pb specimen crack length, a, and notch thickness, t, if applicable,
  - 10.1.5.2 For the sc specimen the crack dimensions a and 2c,
- 10.1.5.3 For the vb 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 Load or displacement rate,
- 10.1.8 Measured inclination of the crack plane as specified in the appropriate annex,
- 10.1.9 Relevant maximum test load,  $P_{\text{max}}$ , as specified in the appropriate annex,

- 10.1.10 Loading diagrams as required,
- 10.1.11 Number of specimens tested and the number of valid tests,
  - 10.1.12 Fracture toughness value with statement of validity,
- 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.

#### 11. Precision and Bias

- 11.1 Precision—The precision of a fracture toughness measurement is a function of the precision and bias of the various measurements of linear dimensions of the test specimen and testing fixtures, and the bias of the load 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.
- 11.2 Bias—There is no accepted "standard" value for the fracture toughness of any material. As discussed in 1.4, 6.1 and 6.2,  $K_{Ipb}$ ,  $K_{Isc}$ , 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 I and Kin Table 2). The results of the last line in Table 2 were generated from a single billet identified as "C."

# 12. Keywords

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

		Krah DATA SHEET			
Material:		Form:	Processing Details:	ig Details:	
Specimen ID:		Test System:	Elastic Mo	Elastic Modulus (GPa):	
Date:		Machine Compliance (m/N):		Mean grain size (10 <sup>-6</sup> m):	
Test Particulars	Data	Test Method Section	ure Test	Data	Test Method Section
Ambient Environment		9.3, 9.9	Crack Length, a (mm)		A2.3.5
Relative Humidity (%RH)		eh 6'6		:	A2.3.5, Fig. 4.
Temperature (°C)		ai/ 6'6	Right, a <sub>0.75</sub> (mm)		A2.3.5, Fig. 4.
		cat	Left, a <sub>0.25</sub> (mm)		A2.3.5, Fig. 4.
Specimen/Crack Plane Orientation		3.2.3 3.2.3	Normalized Crack Length, a/W		A2.3.5
		lan	Final Crack Plane Angle (°)		A2.3.6, Fig. A2.4
Load Configuration (3- or 4-point)		dards	iT 5:/		
Outer span, So (mm)		7.4 /sia	Test Rate (mm/s)		9.7, A2.3.4
		7.4 AS	t		
Specimen dimensions		TN 28	Test Record		A2.3.7, Fig. A2.5
B (mm)		A2.1.1, Figs. 4 & A2.1	Details		
W (mm)		A2.1.1, Figs. 4 & A2.1	da		
Precracking	Data	Test Method Section	Calculation of K <sub>Ipb</sub>	Data	Test Method Section
Notch Dimensions (if used)		<b>e</b> 9 f-4	Maximum Load, P <sub>max</sub> (N)		9.8.1
Notch Length (mm)		A2.1.2.4, Fig. A2.3	Coefficient for Stress Intensity Factor, a (a/W) or f (a/w)		A2.5.2, A2.5.3, A2.5.4
Notch Thickness (mm)		A2.1.2.4, Fig. A2.3			
		£2d53	Fracture Toughness, — K <sub>Iob</sub> (MPa √m)		A2.5
Number of Indents (if used)		A2.3.1			
Indentation Load (N)		42.3.1 d/ast	Crack Stability at Fracture (unstable/stable)		A2.3.7, Fig. A2.5
Fixture groove width (mm)		A2.3.3, Fig. A2.2			
Precracking test rate		42.3.2	Valid K <sub>Ipb</sub> (Y/N)		9.2, A2.3.5, A2.3.6, A2 3 7 A2 6

FIG. 7 Reporting Table for Determination of Fracture Toughness, K<sub>lpb</sub>

497)	С	1	42 <sup>2</sup>
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		T			
Material:		N <sub>Isc</sub> DATA SHEET Form:		Processing Details:	
Specimen ID:	•	Test System:	Elastic Mo	Elastic Modulus (GPa):	
Date:		Machine Compliance (m/N):		Mean grain size (10 <sup>-6</sup> m):	
Test Particulars	Data	Test Method Section	Fracture Test	Data	Test Method Section
Ambient Environment		9.3, 9.9	Crack Dimensions		
Relative Humidity (%RH)		6.6			A3.3.4, Figs. 5 & A3.4
Temperature (°C)		.ai 6.6	c (mm)	A CONTRACTOR OF THE CONTRACTOR	A3.3.4, Figs. 5 & A3.4
Specimen/Crack Plane Orientation		3.2.3	Crack Details (valid crack?)		A3.5.1, A3.5.2, A3.5.3, A3.5.4
		] /sta			
Load Configuration (4-point only)		7.4 andar	Test Rate (N/s)		9.7, A3.3.3
Outer span, So (mm)		ds/	[e		
Inner span, S <sub>i</sub> (mm)		1.4 ± 1.7 ± 1.1 ±	Test Record		
Specimen Dimensions		<b>ne</b> <u>STM</u> /d283	Stable Crack Extension (Y/N)		A3.5.5
B (mm)		A3.1.1, Figs. 5 & The second s	Fractography (SEM/Optical)		X1
Initial (pre-polish) W (mm)		A3.3.2.2, Figs. 5 &	ıda ırd		
Precracking	Data	Test Method Section	Calculation of K <sub>Isc</sub>	Data	Test Method Section
Indent Type		<b>16</b>	Maximum Load, P <sub>max</sub> (N)		9.8.1, A3.3.3
(Knoop or Canted Vickers)		A3.3.1, X3	Coefficients for Stress Intensity Factor		A3.4.1
Indent Diagonal, d (mm)		A3.3.2.1	○ Deepest, Y <sub>d</sub>		A3.4.1.1
Indent Depth, h (mm)		A3.3.2.1	Surface, Y <sub>s</sub>		A3.4.1.2
Post-polish W (mm)		A3.3.2.5, A3.3.2.9	Measured Fracture Toughness,		A3.4.2, A3.5.5
		d/a	K <sub>Isc</sub> (MPa √m)		
Amount removed by polish or hand grinding (mm)		A3.3.2.5, Fig. A3.5	Apparent Fracture Toughness,		A3.4.2, A3.5.5
		:141	K <sub>Isc</sub> * (MPa √m)		
Indentation Load (N)		A3.3.1.2	Valid K $_{ m Isc}$ (Y/N)		9.2, A3.5 , A3.6
		9			

FIG. 8 Reporting Table for Determination of Fracture Toughness, K<sub>Isc</sub>

		K <sub>Ivb</sub> DATA SHEET			
Material:		Form:		Processing Details:	
Specimen ID:		Test System:	Elastic Me	Elastic Modulus (GPa):	
Date:		Machine Compliance (m/N):		Mean grain size (10 <sup>-6</sup> m):	
Test Particulars	Data	Test Method Section	ture Test	Data	Test Method Section
Ambient Environment		9.3, 9.9	Chevron Notch Details		A4.3.1, Figs. 6 & A4.1
Relative Humidity (%RH)		tel 6.6	a <sub>0</sub> (mm)		A4.3.1, Figs. 6 & A4.
Temperature (°C)		6.6	a <sub>11</sub> (mm)		A4.3.1, Figs. 6 & A4.
		cai	a <sub>12</sub> (mm)		A4.3.1, Figs. 6 & A4.
Specimen/Crack Plane Orientation		3.2.3 <u>sol</u>	Notch thickness, t (mm)		A4.4.2, Fig. A4.1
		<b>Do</b>	Notch thickness <0.150 mm at root radius (Y/N)		A4.1.3, A4.3.4
Load Configuration (3- or 4-point)		rds/si	Chevron tip within 0.02 B of specimen center (Y/N)		A4.1.3, A4.3.4
Outer span, S <sub>o</sub> (mm)		AS 4.2	n		
Inner span, S <sub>i</sub> (mm)		7.4 T. 28	Test Rate (mm/s)		9.7, A4.3.3
Specimen dimensions			t:		
B (mm)		A4.1, Figs. 6 & A4.1	Test Record		A4.3.6, Fig. A4.3
W (mm)		A4.1, Figs. 6 & A4.1	Details (stable/unstable)		
Precracking (if used)	Data	Test Method Section	Calculation of K <sub>Ivb</sub>	Data	Test Method Section
Chevron Notch Tip		<b>\</b> lab	Maximum Load, P <sub>max</sub> (N)		9.8.2, Fig. A4.3
in Compression (Y/N)		0-9	Coefficient for Stress		A4.5.1, A4.5.2,
		:YV	Fracture Toughness		A4.5.3, A4.5.4 A4.5.1
		1f2c	K <sub>Ivb</sub> (MPa √m)		
		5.			
Maximum Compressive Load (N)		<b>A4.4.1</b>	Crack Stability at Fracture (Y/N)		A4.3.6, Fig. A4.3
		10			
Number of Load Cycles		/astn:	Valid K <sub>Ivb</sub> (Y/N)		9.2, A4.3.5, A4.3.6, A4.6

FIG. 9 Reporting Table for Determination of Fracture Toughness, K<sub>Ivb</sub>