



Designation: C 1421 – 01b (Reapproved 2007)

# 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 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).

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 E 399, which measure fracture toughness,  $K_{Ic}$ , 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 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 E 399 and may not be interchangeable with each other.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee C28 on Advanced Ceramics and is the direct responsibility of Subcommittee C28.01.

Current edition approved Feb. 1, 2007. Published March 2007. Originally approved in 1999. Last previous edition approved in 2001 as C 1421 - 01b.

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.

Main Body	Section
Scope	1
Referenced Documents	2
Terminology (including definitions, orientation and symbols)	3
Summary of Test Methods	4
Significance and Use	5
Interferences	6
Apparatus	7
Test 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	X3

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.

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

- 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
  - E 337** Test Method for Measuring Humidity with a Psychrometer (the Measurement of Wet- and Dry-Bulb Temperatures)
  - E 399** 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 Ceramics<sup>3</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. (**E 1823**)

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*,  $K [FL^{-3/2}]$ —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**.

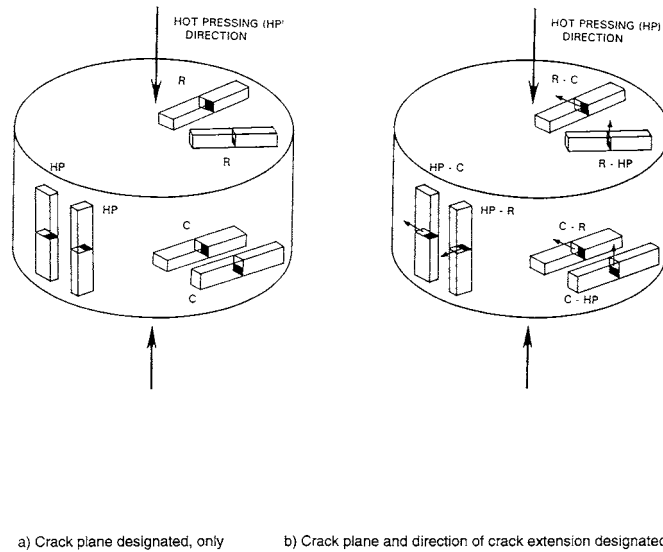
3.2.3.6 Identification of the plane and direction of crack extension is recommended. The plane and direction of crack extension are denoted by a hyphenated code with the first letter(s) representing the direction normal to the crack plane, and the second letter(s) designating the expected direction of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

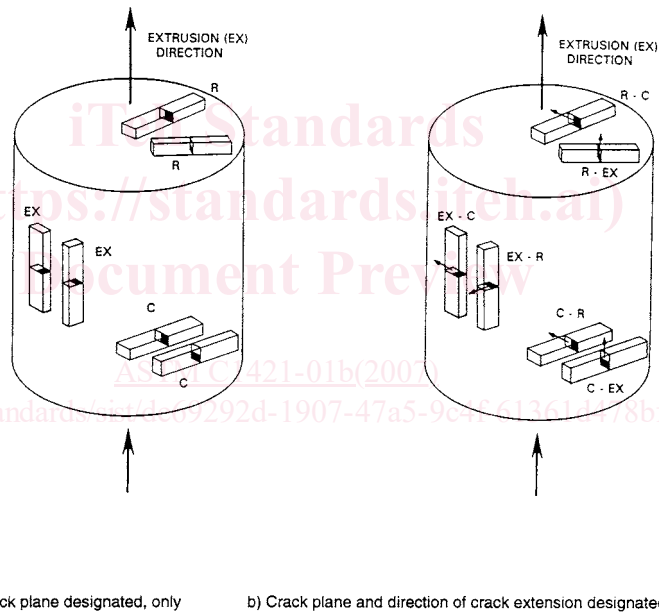
3.2.3.8 Isopressed products, amorphous ceramics, glasses and glass ceramics are often isotropic, and crack plane orientation has little effect on fracture toughness. Nevertheless, the designation of crack plane relative to product geometry is recommended. For example, if the product is isopressed (either

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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



NOTE 1—Precracked beam test specimens are shown as examples. The small arrows denote the direction of crack growth.  
**FIG. 1 Crack Plane Orientation Code for Hot-Pressed Products**



NOTE 1—Precracked beam test specimens are shown as examples. The small arrows denote the direction of crack growth.  
**FIG. 2 Crack Plane Orientation Code for Extruded Products**

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.

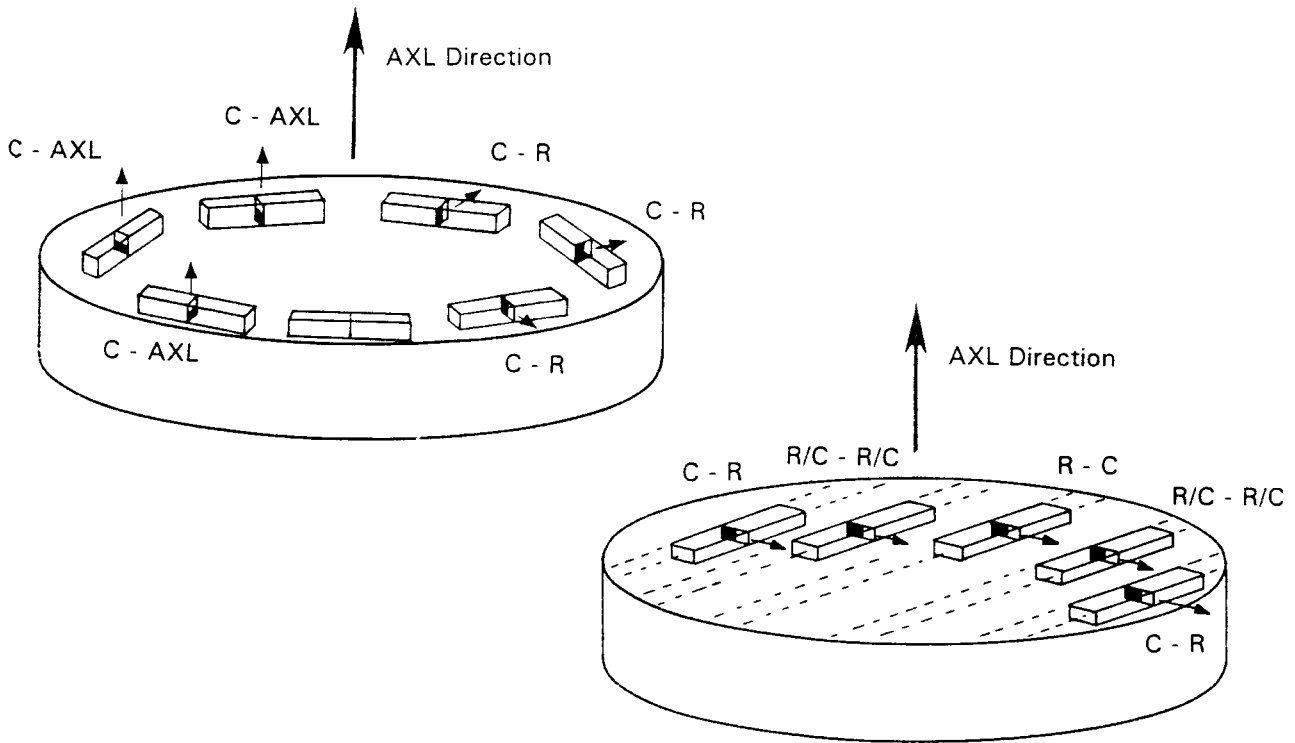
3.2.3.9 If there is no primary product direction, reference axes may be arbitrarily assigned but must be clearly identified.

3.2.4 critical crack size  $[L]$ —in these test methods, the crack size at which maximum 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 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)

3.2.6 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).



- 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 test specimens from the product.

NOTE 2—Precracked beam test 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 Test Specimens with Axial Primary Product Direction

ASTM C1421-01b(2007)

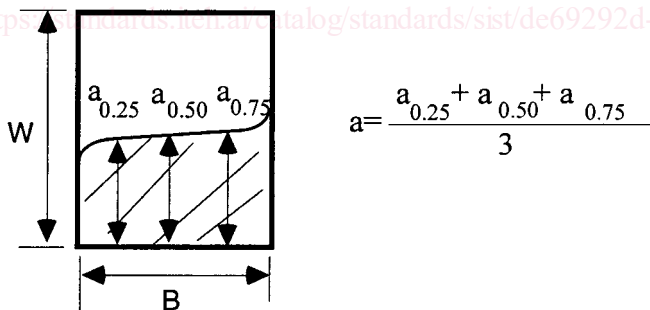


FIG. 4 Cross Section of a pb Test Specimen Showing the Precrack Configuration ( $a_{0.25}$ ,  $a_{0.50}$ ,  $a_{0.75}$  are the Points for Crack Length Measurements)

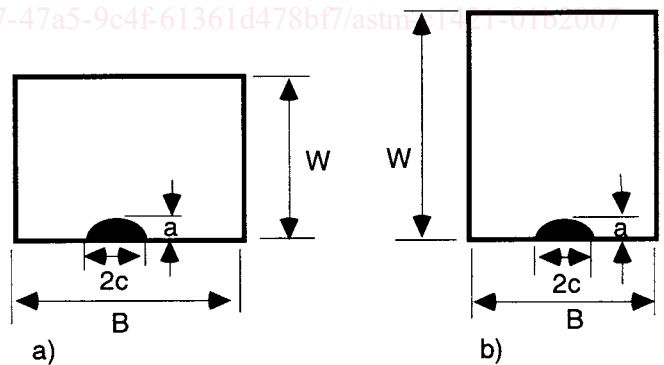


FIG. 5 a and b Cross Section of sc Test Specimens Showing the Precrack Configurations for Two Orientations

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 sudden formation or extension of a crack without catastrophic fracture of the test

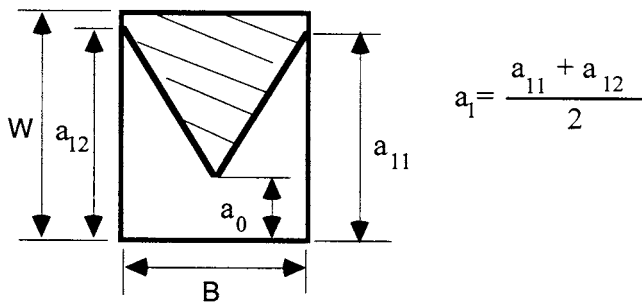


FIG. 6 Cross Section of a vb Test Specimen Showing the Notch Configuration

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. (E 1823)

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

3.2.13.1 *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 *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 *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.

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 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 test 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.

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 flexure, Eq A2.6.

3.3.15  $F$ —indent force, sc method.

3.3.16  $g(a/W)$ —function of the ratio  $a/W$ , pb method, three-point flexure, Eq A2.2 and Eq A2.4.

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 test 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  $K_I$ —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  $L$ —test 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  $P$ —force.

3.3.28  $P_{max}$ —force 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  $S_o$ —outer span, three- or four-point test fixture. Figs. A1.1 and A1.2.

3.3.32  $S_i$ —inner span, four-point test 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 test 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  $Y_{max}$ —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 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)<sup>4</sup> and (2).

**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).

**4.4 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).

**NOTE 3**—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.

## 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>4</sup> 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 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.

**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).

**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 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 accuracy of the testing machine shall be in accordance with Practice E 4.

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

NOTE 5—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.

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 Fig. A1.1, as specified in Test Method C 1161 where 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  $S_o = 20$  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.

7.4.2 The length of each roller shall be at least three times the test specimen dimension, B. The roller diameter shall be  $4.5 \pm 0.5$  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.

7.4.5 A suggested three-point test fixture design is shown in Fig. A1.2. Choose the outer support span,  $S_o$ , such that  $4 \leq \frac{S_o}{W} \leq 10$ , 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 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.

NOTE 7—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  $3.3 \times 10^{-8}$  m/N (including load cell and fixtures) may be required for a typical stable pb test. (See Refs. (8) and (9).)

NOTE 8—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  $4.43 \times 10^{-5}$  m/N (including load cell and fixtures) is adequate for most vb tests.

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.

## 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 (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.

NOTE 9—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 E 399).

8.3 *Test 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 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.

**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  $S_o$ . 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,  $S_o=16$  mm) and  $S_o/W=4.0$  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).

**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 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**—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,
- 10.1.5 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,
- 10.1.13 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 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.

**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



**TABLE 1 Fracture Toughness Values of Sintered Silicon Carbide (Hexoloy SA) in MPa  $\sqrt{m}$** 

(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 $\pm$ 0.20 (3)	2.69 $\pm$ 0.08 (6) <sup>A</sup>	2.62 $\pm$ 0.06 (6) (A config.) 2.68 $\pm$ 0.03 (a) (B config.)	<sup>A,B</sup> using II-UW material, vintage 1985
2.58 $\pm$ 0.08 (4)	2.76 $\pm$ 0.08 (4) <sup>A</sup>	2.61 $\pm$ 0.05 (6) (A config.) 2.46 $\pm$ 0.03 (5) (C config.)	<sup>A,B</sup> using JAS material, vintage 1980
...	3.01 $\pm$ 0.35 (3) <sup>C</sup>	2.91 $\pm$ 0.31 (3) (B config.)	<sup>D</sup>

<sup>A</sup>G.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 press, July 2001

<sup>B</sup>J.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.

<sup>C</sup>This data set may have been susceptible to overestimation of the sc fracture toughness due to the interference of vestigial lateral cracks.

<sup>D</sup>A. Ghosn, M.G. Jenkins, K.W. White, A.S. Kobayashi, and R.C. Bradt, "Elevated-Temperature Fracture Resistance of a Sintered  $\alpha$ -Silicon Carbide," *J. Am. Ceram. Soc.*, 72 [2] pp. 242–247, 1989.

**TABLE 2 Fracture Toughness of Hot-Pressed Silicon Nitride (NC 132) in MPa  $\sqrt{m}$** 

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

Precracked Beam (pb)	Surface Crack in Flexure (sc)	Chevron-Notch (vb)	Ref
...	4.59 $\pm$ 0.37 (107)	4.42 $\pm$ 0.14 (2)	<sup>A</sup>
4.67 $\pm$ 0.3 (7) Stable	4.64 $\pm$ 0.4 (5) <sup>B</sup>	...	<sup>C</sup>
4.50 $\pm$ 0.43 (3) Stable	...	4.85 $\pm$ ? (4)	<sup>D</sup>
4.54 $\pm$ 0.12 (7) Unstable	...	...	<sup>E</sup>
4.19 $\pm$ 0.19 (5) Stable	...	4.84 $\pm$ ? (4)	<sup>F</sup>
...	4.65 $\pm$ 0.10 (?) <sup>B</sup>	...	<sup>G</sup>
...	4.64 $\pm$ 0.25 (4) <sup>B</sup>	...	<sup>H</sup>
...	4.48 $\pm$ 0.07 (4) <sup>B</sup>	...	...
...	4.33 $\pm$ 0.37 (3) <sup>B</sup>	...	...
4.59 $\pm$ 0.12 (11) <sup>I</sup> Valid <sup>J</sup>	4.55 $\pm$ 0.14 (14) <sup>I</sup> Valid <sup>J</sup>	4.60 $\pm$ 0.13 (8) <sup>I</sup> Valid <sup>J</sup>	<sup>K</sup>

<sup>A</sup>G.D. Quinn, J.J. Kübler, and R.J. Gettings, "Fracture Toughness of Advanced Ceramics by the Surface Crack in Flexure (SCF) Method: A VAMAS Round Robin," VAMAS Report # 17, National Institute of Standards and Technology, Gaithersburg, MD, June 1994.

<sup>B</sup>Annealed to remove indentation residual stresses. Note that although annealing to remove residual stresses is not allowed for the sc method in this standard, data are included here for illustrative purposes.

<sup>C</sup>V. Tikare and S.R. Choi, "Combined Mode I and Mode II Fracture of Monolithic Ceramics," *J. Am. Ceram. Soc.*, 76 [9], pp. 2265–2272, 1993.

<sup>D</sup>J.A. Salem, J.L. Shannon, Jr., and M.G. Jenkins, "Some Observations in Fracture Toughness and Fatigue Testing with Chevron-Notched Specimen," in *Chevron Notch Fracture Test Experience: Metals and Non-Metals*, ASTM STP 1172, eds. K.R. Brown and F.I. Baratta, ASTM, West Conshohocken, PA, pp 9–25, 1992.

<sup>E</sup>I. Bar-On, F.I. Baratta, and K. Cho, "Crack Stability and Its Effect on Fracture Toughness of Hot-Pressed Silicon Nitride Beam Specimens," *J. Am. Ceram. Soc.*, Vol 79 [9], pp. 2300–2308, 1996.

<sup>F</sup>R.T. Bubsey, J.L. Shannon, Jr., and D. Munz, "Development of Plane Strain Fracture Toughness Test for Ceramics Using Chevron Notched Specimens," in *Ceramics for High Performance Applications III, Reliability*, eds. E.M. Lenoë, R.N. Katz, and J.J. Burke, Plenum, NY, pp. 753–771, 1983.

<sup>G</sup>J.J. Petrovic, L.A. Jacobson, P.K. Talty, and A.K. Vasudevan, "Controlled Surface Flaws in Hot-Pressed Si<sub>3</sub>N<sub>4</sub>," *J. Am. Ceram. Soc.*, 58 [3–4], pp. 113–116, 1975.

<sup>H</sup>G.D. Quinn and J.B. Quinn, "Slow Crack Growth in Hot-Pressed Silicon Nitride," in *Fracture Mechanics of Ceramics*, Vol 6, eds. R.C. Bradt, A.G. Evans, D.P.H. Hasselman, F.F. Lange, Plenum, NY pp. 603–636, 1983.

<sup>I</sup>Single Billet C

<sup>J</sup>Valid tests per the validity requirements of 9.2 of this test method.

<sup>K</sup>G.D. Quinn, J.A. Salem, I. Bar-On, and M.G. Jenkins, "The New ASTM Fracture Toughness of Advanced Ceramics: PS070–97," *Ceramic Engineering and Science Proceedings*, Vol 19, No 3, pp. 565–578, 1998.

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 $\sqrt{m}$  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 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

### K<sub>Ipb</sub> DATA SHEET

Material:

Form:

Processing Details:

Test Specimen ID:

Test System:

Elastic Modulus (GPa):

IT

Date:

Test 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 Length, a (mm)		A2.3.5
Relative Humidity (%RH)		9.9	Center, a <sub>0.50</sub> (mm)		A2.3.5, Fig. 4.
Temperature (°C)		9.9	Right, a <sub>0.75</sub> (mm)		A2.3.5, Fig. 4.
			Left, a <sub>0.25</sub> (mm)		A2.3.5, Fig. 4.
Test Specimen/Crack Plane Orientation		3.2.3	Normalized Crack Length, a/W		A2.3.5
			Final Crack Plane Angle (°)		A2.3.6, Fig. A2.4
Flexure Configuration (3- or 4-point)		7.4			
Outer span, S <sub>o</sub> (mm)		7.4	Test Rate (mm/s)		9.7, A2.3.4
Inner span, S <sub>i</sub> (mm)		7.4			
Test Specimen Dimensions			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			
Precracking	Data	Test Method Section	Calculation of K <sub>Ipb</sub>	Data	Test Method Section
Notch Dimensions (if used)			Maximum Force, P <sub>max</sub> (N)		9.8.1
Notch Length (mm)		A2.1.2.4, Fig. A2.3	Coefficient for Stress Intensity Factor, g (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			
			Fracture Toughness, K <sub>Ipb</sub> (MPa √m)		A2.5
Number of Indents (if used)		A2.3.1			
Indentation Load (N)		A2.3.1	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		A2.3.2	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>Ipb</sub>

### K<sub>Isc</sub> DATA SHEET

Material:

Form:

Processing Details:

Test Specimen ID:

Test System:

Elastic Modulus (GPa):

Date:

Test 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)		9.9	a (mm)		A3.3.4, Figs. 5 & A3.4
Temperature (°C)		9.9	c (mm)		A3.3.4, Figs. 5 & A3.4
Test Specimen/Crack Plane Orientation		3.2.3	Crack Details (valid crack?)		A3.5.1, A3.5.2, A3.5.3, A3.5.4
Flexure Configuration (4-point only)		7.4	Test Rate (N/s)		9.7, A3.3.3
Outer span, S <sub>o</sub> (mm)		7.4			
Inner span, S <sub>i</sub> (mm)		7.4	Test Record		
Test Specimen Dimensions			Stable Crack Extension (Y/N)		A3.5.5
B (mm)		A3.1.1, Figs. 5 & A3.1	Fractography (SEM/Optical)		X1
Initial (pre-polish) W (mm)		A3.3.2.2, Figs. 5 & A3.1			
Precracking	Data	Test Method Section	Calculation of K <sub>Isc</sub>	Data	Test Method Section
Indent Type			Maximum Force, 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, K <sub>Isc</sub> (MPa √m)		A3.4.2, A3.5.5
Amount removed by polish or hand grinding (mm)		A3.3.2.5, Fig. A3.5	Apparent Fracture Toughness, K <sub>Isc</sub> * (MPa √m)		A3.4.2, A3.5.5
Indentation Force (N)		A3.3.1.2	Valid K <sub>Isc</sub> (Y/N)		9.2, A3.5, A3.6

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