Designation: C1421 – 10

Standard Test Methods for Determination of Fracture Toughness of Advanced Ceramics at Ambient Temperature[1]

This standard is issued under the fixed designation C1421; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

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

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

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

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

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

1.5 This standard begins with a main body that provides information on fracture toughness testing in general. It is followed by annexes and appendices with specific information for the particular test methods.

1.6 Values expressed in these test methods are in accordance with the International System of Units (SI) and Practice IEEE/ASTM SI 10.

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

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C1161 Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature

C1322 Practice for Fractography and Characterization of Fracture Origins in Advanced Ceramics

E4 Practices for Force Verification of Testing Machines

E112 Test Methods for Determining Average Grain Size

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

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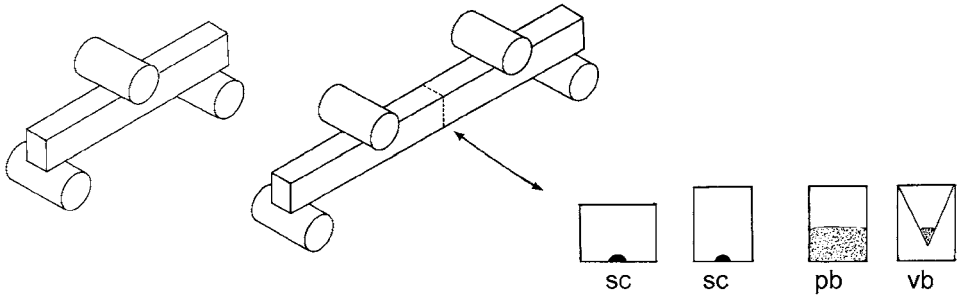
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.



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

FIG. 1 The Three Test Methods

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E337 Test Method for Measuring Humidity with a Psychrometer (the Measurement of Wet- and Dry-Bulb Temperatures)
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E740 Practice for Fracture Testing with Surface-Crack Tension Specimens
E1823 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[3]

3. Terminology

3.1 *Definitions:*

3.1.1 The terms described in Terminology E1823 are applicable to these test methods. Appropriate sources for each definition are provided after each definition in parentheses.

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

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

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

3.1.5 *stress-intensity factor, K* $[FL^{-3/2}]$—the magnitude of the ideal-crack-tip stress field (stress field singularity) for a particular mode in a homogeneous, linear-elastic body. **(E1823)**

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 *critical crack size [L]*—The crack size at which maximum force and catastrophic fracture occur in the precracked beam and the surface crack in flexure configurations. In the chevron-notched test specimen 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.

3.2.4 *four-point - ¼ 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) **(C1161)**

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

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

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

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

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

3.2.10 *pop-in*—The sudden formation or extension of a crack without catastrophic fracture of the test specimen, apparent from a force drop in the applied force-displacement

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

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

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

3.2.13 *three-point flexure*—flexure configuration where a beam test specimen is loaded at a location midway between two support bearings (see Fig. A1.2) **(C1161)**

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

3.3 *Symbols:*

3.3.1 a—crack depth, crack length, crack size.

3.3.2 a_o—chevron tip dimension, vb method, Fig. A4.1.

3.3.3 a_1—chevron dimension, vb method, $(a_1 = (a_{11} + a_{12})/2)$, Fig. A4.1

3.3.4 a_{11}—chevron dimension, vb method, Fig. A4.1.

3.3.5 a_{12}—chevron dimension, vb method, Fig. A4.1.

3.3.6 $a_{0.25}$—crack length measured at 0.25B, pb method, Fig. A4.2.

3.3.7 $a_{0.50}$—crack length measured at 0.5B, pb method, Fig. A4.2.

3.3.8 $a_{0.75}$—crack length measured at 0.75B, pb method, Fig. A4.2.

3.3.9 *a/W*—normalized crack size.

3.3.10 B—the side to side dimension of the test specimen perpendicular to the crack length (depth) as shown in Fig. A2.4, Fig. A3.7, and Fig. A4.1.

3.3.11 c—crack half width, sc method, Fig. A3.7.

3.3.12 d—length of long diagonal for a Knoop indent, length of a diagonal for a Vickers indent, sc method.

3.3.13 E—elastic modulus.

3.3.14 *f(a/W)*—function of the ratio a/W, pb method, four-point flexure, Eq A2.6.

3.3.15 F—indent force, sc method.

3.3.16 F_C—chamfer correction factor, sc method

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

3.3.18 h—depth of Knoop or Vickers indent, sc method, Eq A3.1.

3.3.19 $H_1(a/c, a/W)$—a polynomial in the stress intensity factor coefficient, for the precrack periphery where it intersects the test specimen surface, sc method, Eq A3.7.

3.3.20 $H_2(a/c, a/W)$—a polynomial in the stress intensity factor coefficient, for the deepest part of a surface crack, sc method, see Eq A3.5.

3.3.21 K_I—stress intensity factor, Mode I.

3.3.22 K_{Ic}—fracture toughness, critical stress intensity factor, Mode I.

3.3.23 K_{Ipb}—fracture toughness, pb method, Eq A2.1 and Eq A2.3.

3.3.24 K_{Isc}—fracture toughness, sc method, Eq A3.9.

3.3.25 K_{Ivb}—fracture toughness, vb method, Eq A4.1.

3.3.26 L—test specimen length, Fig. A2.1and Fig. A3.1.

3.3.27 L_1, L_2—precracking fixture dimensions, pb method, Fig. A2.2.

3.3.28 *M(a/c, a/W)*—a polynomial in the stress intensity factor coefficient, sc method, see Eq A3.4.

3.3.29 P—force.

3.3.30 P_{max}—force maximum.

3.3.31 *Q(a/c)*—a polynomial function of the surface crack ellipticity, sc method, Eq A3.3.

3.3.32 *S(a/c, a/W)*—factor in the stress intensity factor coefficient, sc method, Eq A3.8.

3.3.33 S_o—outer span, three- or four-point test fixture. Figs. A1.1 and A1.2.

3.3.34 S_i—inner span, four-point test fixture, Fig. A1.1.

3.3.35 t—notch thickness, pb and vb method, Fig. A2.3 and Fig. A4.1.

3.3.36 W—the top to bottom dimension of the test specimen parallel to the crack length (depth) as shown in A2.4, A3.7, and A4.1.

3.3.37 Y—stress intensity factor coefficient.

3.3.38 Y^*—stress intensity factor coefficient for vb method.

3.3.39 Y_{max}—maximum stress intensity factor coefficient occurring around the periphery of an assumed semi-elliptical precrack, sc method

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

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

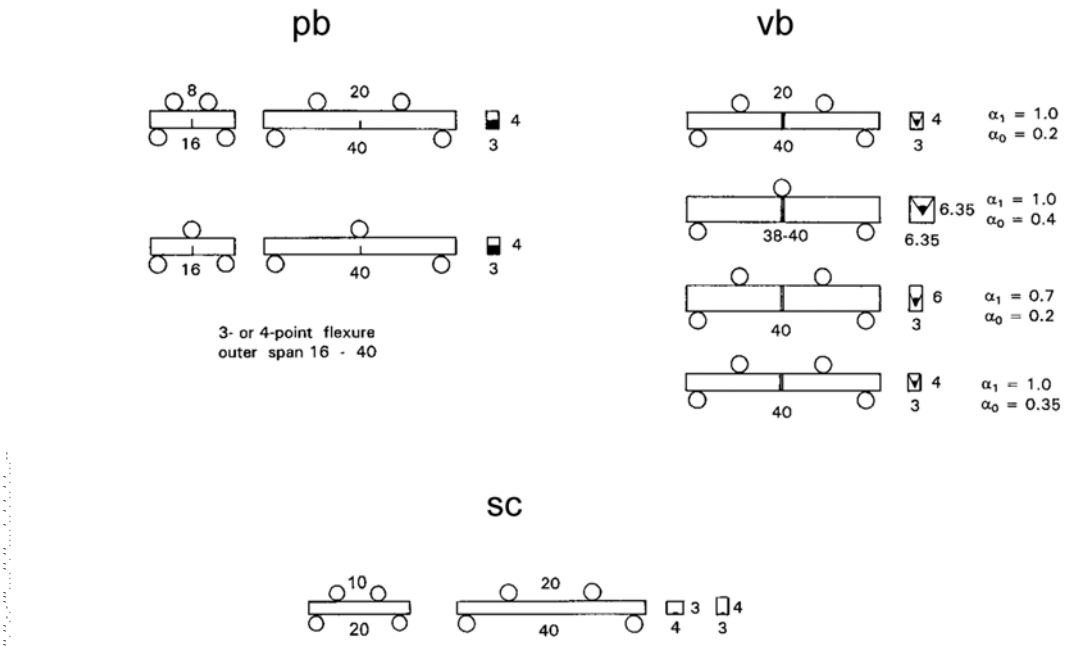
3.3.42 Y_s—stress intensity factor coefficient at the intersection of the surface crack with the test specimen surface, sc method, Eq A3.6

4. Summary of Test Methods

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

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

4.3 *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 or Knoop



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

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

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

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

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

NOTE 2—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 Fracture toughness, K_{Ic}, is a measure of the resistance to crack extension in a brittle material. These test methods may be used for material development, material comparison, quality assessment, and characterization.

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

6. Interferences

6.1 *R-curve*—The microstructural features of advanced ceramics can cause rising R-curve behavior. For such materials the three test methods are expected to result in different fracture toughness values. These differences are due to the amount of crack extension prior to the relevant maximum test force, P_{max}, 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 the same. NIST Standard Reference Material 2100 has a flat R-curve and $K_{Ipb} = K_{Isc} = K_{Ivb}$.

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

6.3 *Stability*—This standard permits measurements of fracture toughness whereby the crack propagates unstably (sc and pb methods) or stably (sc, pb, vb). The stiffness of the test set-up can affect whether the crack grows stably or unstably. There is limited data that suggests a stably propagating crack may give a slightly lower fracture toughness value than an unstably propagating crack (1-3).

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

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

7. Apparatus

7.1 *Testing*—Use a testing machine that has provisions for autographic recording of force applied to the test specimen versus either test specimen centerline deflection or time. The force accuracy of the testing machine shall be in accordance with Practice E4.

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

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

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

7.4 *Fixtures*—The pb and vb test specimens may be tested in either three-point or four-point fixtures. Annex A2 and Annex A3 give the recommended span sizes for these two methods, respectively. sc test specimens shall only be tested in four-point fixtures. Bend fixtures designed for flexural strength testing in accordance with Test Method C1161 are suitable, but this test method allows spans and configurations not in C1161. A bridge precracking fixture is also necessary for the pb method. It is described in Annex A2.

NOTE 4—Hereafter in this document the term four-point flexure will refer to the specific case of ¼-(that is, quarter) point flexure.

7.4.1 The four-point test fixture (see Fig. A1.1) for the pb, vb, or sc methods shall conform to the general fixture requirements of Test Method C1161. The recommended outer and inner spans are S_o = 40 mm and S_i = 20 mm, respectively, but this standard allows other span sizes provided that the minimum outer and inner spans shall be S_o = 20 mm and S_i = 10 mm, respectively. The outer rollers shall be free to roll outwards and the inner rollers shall be free to roll inwards. Place the rollers initially against their stops and hold them in position by low-tension springs or rubber bands or magnets. Roller pins shall have a hardness of 40 Rockwell C or greater.

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

7.4.3 If the test specimen parallelism requirements set forth in Fig. A2.1 and Fig. A3.1 are not met, use a fully-articulating fixture as described in C1161.

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

7.4.5 A three-point test fixture (see Fig. A1.2) may be used for the vb and pb methods. For the pb method, use an outer

span, S_o, between 16 and 40 mm. Since W = 4 mm (the top to bottom dimension of the test specimen parallel to the crack length), then the fixture span to specimen size ratio is: $4 \leq \frac{S_o}{W} \leq 10$. For the vb method, W can range from 4 mm to 6.35 mm depending on the specimen type in Annex A4. Choose an outer span, S_o, such that $4 \leq \frac{S_o}{W} \leq 10$. The outer two rollers shall be free to roll outwards to minimize friction effects. The middle flexure roller shall be fixed. Alternatively, a rounded knife edge with diameter in accordance with 7.4.2 may be used in place of the middle roller.

NOTE 5—A stiff test system with displacement control and a stiff load train may be required to obtain stable crack extension for the vb test. Stable crack extension is essential for a valid vb test. A test system compliance of less than or equal to 4.43×10^{-5} m/N (including load cell and fixtures) is adequate for most vb tests. Stable crack extension is not required for the pb test, but if it is desired, then a stiff load train may be required. See Refs. (8) and (9).

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

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

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

8. Test Specimen Configurations, Dimensions and Preparation

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

NOTE 6—A typical "plastic" (or deformation) zone, if such exists, is no greater than a fraction of a micrometer in most ceramics, thus the specified sizes are large enough to meet generally-accepted plane strain requirements at the crack tip from a plasticity viewpoint.

9. General Procedures for Test Methods and Calculations

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

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

9.2.1 A valid test shall meet the following general requirements.

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 E4 (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.

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

9.4 *R-curve*—When rising R-curve behavior is to be documented, two different test methods with different amounts of stable crack extension should be used and the results compared. The pb and sc tests typically have less stable crack extension than the vb test.

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

9.6 *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.

9.7 *Humidity and Temperature*—Measure the temperature and humidity according to Test Method E337.

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 E112 or other appropriate method,

10.1.4 Environment of test, relative humidity, temperature,

10.1.5 Test specimen dimensions: B and W,

10.1.5.1 For the pb test specimen crack length, a, and notch thickness, t, if applicable,

10.1.5.2 For the sc test specimen the crack dimensions a and $2c$,

10.1.5.3 For the vb test specimen the notch parameters, a_0 and a_{11} and a_{12} and the notch thickness, t,

10.1.6 Test fixture specifics,

10.1.6.1 Whether the test was in three- or four-point flexure,

10.1.6.2 Outer span, S_o, and inner span (if applicable), S_i,

10.1.7 Applied force or displacement rate,

10.1.8 Measured inclination of the crack plane as specified in the appropriate annex,

10.1.9 Relevant maximum test force, P_{max}, as specified in the appropriate annex,

10.1.10 Testing diagrams (for example, applied force vs. displacement) as required,

10.1.11 Number of test specimens tested and the number of valid tests,

10.1.12 Fracture toughness values for each valid test with a statement confirming that all tests were indeed valid,

10.1.13 Additional information as required in the appropriate annex, and

10.2 Mean and standard deviation of the fracture toughness for each test method used.

10.3 Crack plane and direction of crack propagation as appropriate (see Appendix X5).

11. Precision and Bias

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

11.2 *Bias*—Standard Reference Material (SRM) 2100 from the National Institute of Standards and Technology may be used to check for laboratory test result bias. The laboratory average value may be compared to the certified reference value of fracture toughness of 4.57 MPa$\sqrt{}$m ± 0.11 MPa$\sqrt{}$m (or 2.3 %) at a 95 % confidence level. SRM 2100 is a set of five silicon nitride beam test specimens. Identical results are obtained with the three test methods in this standard when used with SRM 2100.

11.3 *Variation in Results with Test Method for Other Materials*—As discussed in 1.4, 6.1 and 6.2, for some materials K_{Ipb}, K_{Isc}, and K_{Ivb} values may differ from each other (for example, **(15)**). Nevertheless, a comparison of test results obtained by the three different methods is instructive. Such comparisons are shown in Table 1. The experimental procedures used in the studies cited in Table 1 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).

12. Keywords

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

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

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

Precracked Beam (pb)	Surface Crack in Flexure (sc)	Chevron-Notch (vb)	Ref
2.54 ± 0.20 (3)	2.69 ± 0.08 (6)[A]	2.62 ± 0.06 (6) (A config.) 2.68 ± 0.03 (2) (B config.)	[A,B] using II-UW material, vintage 1985
2.58 ± 0.08 (4)	2.76 ± 0.08 (4)[A]	2.61 ± 0.05 (6) (A config.) 2.46 ± 0.03 (5) (C config.)	[A,B] using JAS material, vintage 1980
...	3.01 ± 0.35 (3)[C]	2.91 ± 0.31 (3) (B config.)	[D]

[A]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.*85 [4] pp. 873 – 880, 2002.
[B]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.
[C]This data set may have been susceptible to overestimation of the sc fracture toughness due to the interference of vestigial lateral cracks.
[D]A. Ghosn, M. G. Jenkins, K. W. White, A. S. Kobayashi, and R. C. Bradt, "Elevated-Temperature Fracture Resistance of a Sintered α-Silicon Carbide," *J. Am. Ceram. Soc.*, 72 [2] pp. 242–247, 1989.

ANNEXES

(Mandatory Information)

A1. SUGGESTED TEST FIXTURE SCHEMATICS

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

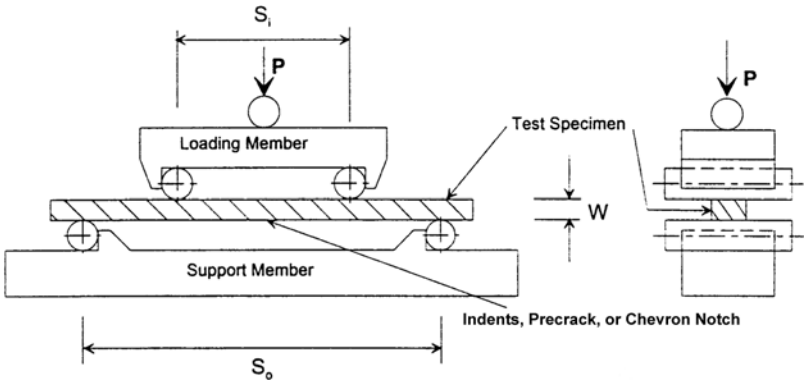


FIG. A1.1 Four-point test fixture schematic which illustrates the general requirements for a semi-articulating fixture.

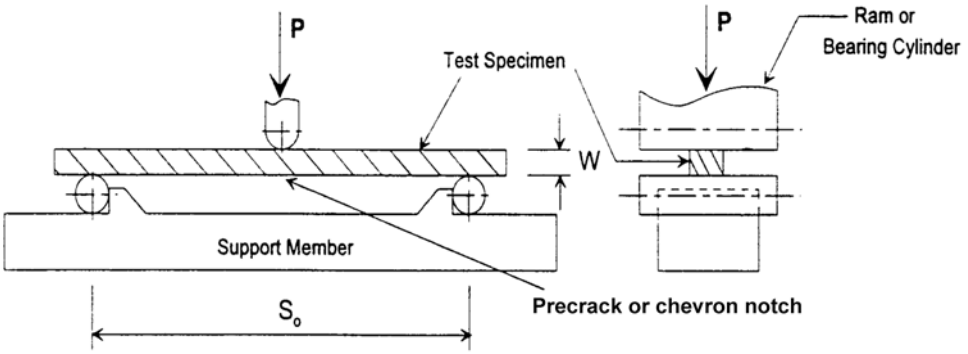


FIG. A1.2 Three-point test fixture schematic which illustrates the general requirements of the test fixture.

A2. PROCEDURES AND SPECIAL REQUIREMENTS FOR THE PRECRACKED BEAM METHOD

A2.1 Test Specimen

A2.1.1 *Test Specimen Size*—The test specimen shall be 3 by 4 mm in cross section with the tolerances shown in Fig. A2.1. The test specimen may or may not contain a saw-cut notch. For both four-point and three-point flexure tests the length shall be at least 20 mm but not more than 50 mm. Test specimens of larger cross section can be tested as long as the proportions given in Fig. A2.1 are maintained.

A2.1.2 *Test Specimen Preparation*—Test specimens prepared in accordance with the Procedure of Test Method C1161, test specimen Type B, are suitable as summarized in the following paragraphs, A2.1.2.1-A2.1.2.3. Alternative procedures may be utilized provided that unwanted machining damage and residual stresses are minimized. Report any alternative test specimen preparation procedure in the test report.

A2.1.2.1 All grinding shall be done with an ample supply of appropriate filtered coolant to keep workpiece and wheel constantly flooded and particles flushed. Grinding shall be in at least two stages, ranging from coarse to fine rates of material removal. All machining shall be in the surface grinding mode parallel to the test specimen long axis. The stock removal rate shall not exceed 0.02 mm per pass to the last 0.06 mm per face.

A2.1.2.2 Perform finish grinding with a diamond-grit wheel of 320 grit or finer. No less than 0.06 mm per face shall be removed during the final finishing phase, and at a rate of not more than 0.002 mm per pass.

A2.1.2.3 The two end faces need not be precision machined. The four long edges shall be chamfered at 45° a distance of 0.12±0.03 mm, or alternatively, they may be rounded with a radius of 0.15 ± 0.05 mm as shown in Fig. A2.1. Edge finishing shall be comparable to that applied to the test specimen surfaces. In particular, the direction of the machining shall be parallel to the test specimen long axis.

A2.1.2.4 The notch, if used, should be made in the 3-mm face, should be less than 0.10 mm in thickness, and should have a length of $0.12 \leq a/W \leq 0.30$.

A2.1.3 It is recommended that at least ten test specimens be prepared. This will provide test specimens for practice tests to determine the best precracking parameters. It will also provide make-up test specimens for unsuccessful or invalid tests so as to meet the requirements of 9.1 and 9.2.

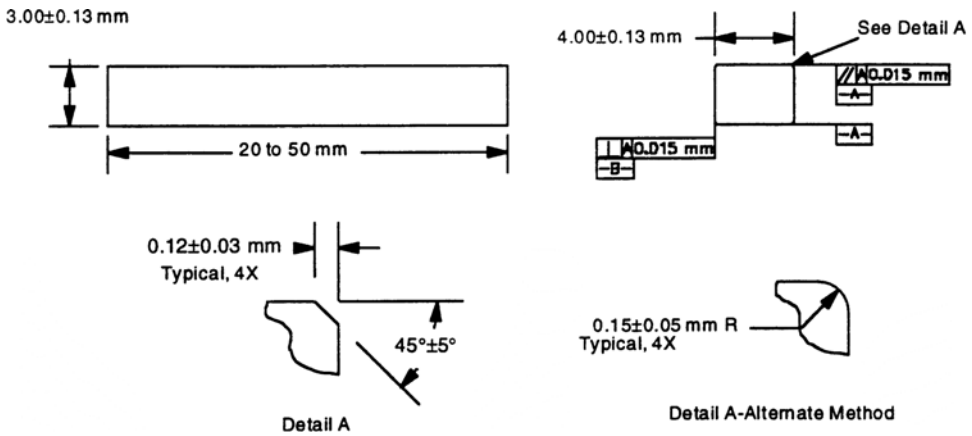


FIG. A2.1 Dimensions of Rectangular Beam

A2.1.4 Measure the cross section dimensions B and W to within 0.002 mm near the middle of the test piece.

A2.2 Apparatus

A2.2.1 *General*—This fracture test is conducted in either three- or four-point flexure. A displacement measurement (or alternative) is required for fracture testing in order to detect signs of crack extension.

A2.2.2 *Bridge Precracking Fixture*—The configuration used for precracking is different from that used for the actual fracture test. A bridge compression fixture is used to create a precrack from an indentation crack or from a sawed notch. The fixture consists of a square support lower plate with a center groove (which is bridged by the test specimen) and a top pusher plate with a bonded pusher plate insert (for example, silicon nitride). The lengths of both plates (L_1 in Fig. A2.2) are equal to each other and are less than or equal to 18 mm. The surfaces that contact the test specimen are of a material with an elastic modulus greater than 300 GPa. The support plate can have several grooves (L_2 in Fig. A2.2) ranging from 2 to 6 mm in width. Alternatively, several parts, each with a different groove width can be used. A fixture design is shown in Fig. A2.2. The support and pusher plates shall be parallel within 0.01 mm. Alternatively, a self-aligning fixture can be used.

A2.2.3 *Fracture Test Fixture*—The general principles of the four-and three-point test fixture are detailed in 7.4 and illustrated in Fig. A1.1 and Fig. A1.2, respectively. For three-point flexure, choose the outer support span such that $4 \leq \frac{S_o}{W} \leq 10$.

A2.2.3.1 For four-point flexure, 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. Seat the displacement indicator (if used) close to the crack plane. Alternatively, use actuator (or crosshead) displacement (stroke), back-face strain, or a time sweep.

A2.2.3.2 For three-point flexure, 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 if used. Alternatively, monitor actuator (or crosshead) displacement, back-face strain, or a time sweep.

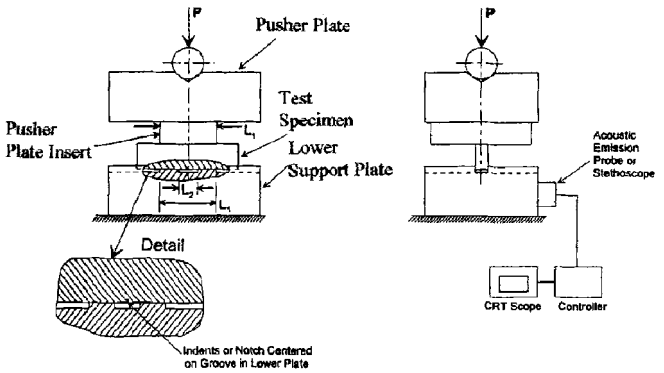


FIG. A2.2 Suggestion for Bridge Compression Fixture (16)

NOTE A2.1—For short spans (for example, S_o = 16 mm) and S_o/W = 4.0 in three-point flexure, 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 (11, 12) .

A2.2.3.3 This method permits either unstable or stable crack extension during the fracture test. When the critical stress intensity, K_{Ipb}, is reached, the crack propagates unstably through the test piece. This is acceptable and the normal way this test method is performed. If stable extension is desired, extra attention to the test setup is needed and very stiff test fixtures and load train may be necessary. The stability (that is, the tendency to obtain stable crack extension) of the test setup is affected not only by the test system compliance (see Note 7) but also by the test specimen dimensions, the S_o/W ratio, and the elastic modulus of the material (8, 9). The degree of stability can be detected easily with back-face strain.

NOTE A2.2—There is a limited amount of data indicating unstable tests may result in slightly greater fracture toughness values than those from tests with stable crack extension (8, 9). If stable crack extension cannot be obtained with four-point flexure, it may be possible to obtain stable crack extension by using a three-point flexure configuration in a stiff test setup. Nonlinearity of the initial part of the applied force-displacement curve (sometimes called "windup") is usually an artifact of the test setup and may not be indicative of material behavior. This type of nonlinearity does not contribute directly to instability unless such nonlinearity extends to the region of maximum force.

A2.3 Procedure

A2.3.1 *Preparation of Crack Starter*—Either the machined notch (Fig. A2.3a), or one or more Vickers or Knoop indentations, (Fig. A2.3b) act as the crack starter. For a test specimen without a notch, create a Vickers indentation in the middle of the surface of the 3-mm face (Fig. A2.3b). Additional indentations can be placed on both sides of the first indentation, aligned in the same plane and perpendicular to the longitudinal axis of the test specimen, as shown in Fig. A2.3b. One of the diagonals of each of the indentations shall be aligned parallel to the test specimen length. The indentation force shall not exceed 100 N. While an indentation crack is physically necessary for subsequent generation of a pop-in crack, cracks emanating from the corners of the indentation may or may not be visible depending on the characteristics and finish of the test material. Alternatively, a Knoop indentation may also be used as a crack starter in which case, the long axis of the indentation shall be perpendicular to the longitudinal axis of the test specimen. If, for a particular test material, a pop-in crack does not form from the indent produced by the 100 N indentation, then it may be necessary to first form a saw notch as a crack starter.

NOTE A2.3—The 100 N indentation force limit is intended to minimize

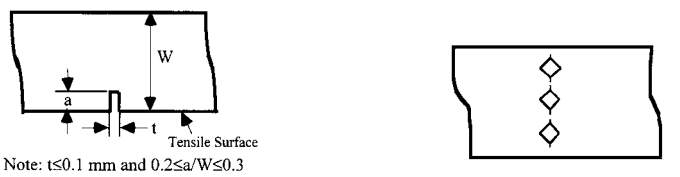


FIG. A2.3 Precracked Beam Precracking Arrangement

potential residual tensile stresses which could influence the fracture results. If residual stresses from the indentation are suspected to have affected the fracture results, the indentations may be removed by polishing, hand grinding or grinding after the precrack has been formed (A2.3.2). Annealing may be used provided that the crack tip is not blunted nor the crack tip/planes healed.

A2.3.2 *Formation of Precrack*—Thoroughly clean the test specimen and contacting faces of the bridge compression fixture. Place the test specimen in the compression fixture with the surface containing the notch or indent(s) over the groove and the notch or indent(s) centered between the edges of the groove. Load the test specimen in the compression fixture at rates less than 1000 N/s until a distinct pop-in sound is heard and/or until a pop-in precrack is seen. At high force rates it may not be possible to discern the force drop in the applied force-displacement curve as discussed in 3.2.10. A stethoscope or other acoustic transducer can also be used to detect the pop-in sound. A traveling microscope is also recommended to view the pop-in crack as the pop-in sound is not always discernible. In some materials it is difficult to see a precrack on the side of the test specimens. Lapping of the side surface or use of a dye penetrant, or both, (see A2.3.2.1) can help delineate the crack. Stop loading immediately after pop-in. Measure the length of the pop-in crack on both side surfaces. The precrack length should be between 0.35 and 0.60*W*.

NOTE A2.4—For materials with a rising R-curve the K_{Ipb} value might be artificially high if the precrack is not stopped immediately after pop-in. The force rate during pop-in may influence the crack/microstructure interaction and may affect the result.

NOTE A2.5—**Caution:** Use care not to overload the testing machine or load cell.

A2.3.2.1 A drop of the dye penetrant can be placed on indentations or saw notch. Upon formation of the precrack, the penetrant will be drawn into the crack and will show on the side surface of the test specimen upon unloading.

NOTE A2.6—**Caution:** Use care to ensure that dye penetrants are dry (for example, by heating the specimen) or do not promote corrosion or slow crack growth, prior to fracture testing to preclude undesired slow crack growth or undesired crack face bonding.

A2.3.3 *Choice of Groove*—The pop-in precrack length is a result of the selected indent force and groove size of the compression fixture. These two parameters need to be determined by trial and error. It has been shown that the pop-in precrack length decreases with increasing indent force and with decreasing groove (span) size **(16, 17)**.

A2.3.4 *Fracture Test*—Insert the test specimen into the flexure fixture. Align the test specimen so that it is centered directly below the axis of the force application. Align the tip of the crack with the centerline of the middle roller in the three-point flexure fixture within 0.5 mm or within 1.0 mm of the midpoint between the two inner rollers, S_i, of the four-point flexure fixture. Seat the displacement indicator (if used) close to the crack plane. Alternatively, monitor actuator (or crosshead) displacement (stroke), back-face strain, or a time sweep. Test the test specimen in actuator displacement (stroke) control at a rate in agreement with 9.6. Record applied force versus displacement or alternative (for example, actuator displacement (stroke), load-point displacement, displacement of the test specimen at the crack plane), back-face strain **(10)** or time.

NOTE A2.7—Generally, actuator displacement (stroke) rates of 0.0005 to 0.01 mm/s for test specimens with a 3 × 4 mm cross section provide stress intensity factor rates in accordance with 9.6.

NOTE A2.8—Actuator displacement (stroke) 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)**.

NOTE A2.9—The requirement for centering the test specimen is much easier to fulfill for a four-point flexure test **(18)**. A three-point flexure test requires that the crack plane be centered accurately in the test fixture.

A2.3.5 *Post Test Measurements*—Fractographically measure the crack length after fracture to the nearest 1 % of W at a magnification greater than or equal to ×20 at the following three positions: at the center of the precrack front and midway between the center of the crack front and the end of the crack front on each surface of the test specimen (Fig. A2.4). Use the

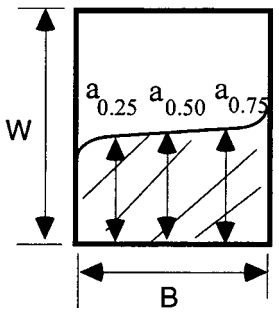


FIG. A2.4 pb Test Specimen Showing the Precrack Configuration ($a_{0.25}$, $a_{0.50}$, $a_{0.75}$ are the Points for Crack Length Measurements)

average of these three measurements to calculate K_{Ipb}. The difference between the average crack length and any precrack length measurement shall be less than 10 %. The average precrack length, a, shall be within the following range: $0.35W \leq a \leq 0.60W$. If the crack was started from a notch, the precrack length, a, shall also be longer than the sum of the notch length and one notch thickness.

A2.3.6 The plane of the final crack measured from the tip of the precrack shall be parallel to both the test specimen dimensions *B* and *W* within ± 5° for three-point flexure and within ±10° for four-point flexure, as illustrated in Fig. A2.5.

A2.3.7 Inspect the applied force-displacement curves. As illustrated in Fig. A2.6, the applied force-displacement curves can indicate a) unstable crack extension (Fig. A2.6a), pop-in (or crack jump) behavior (stable) (Fig. A2.6b), or smooth stable crack extension (Fig. A2.6c). Unstable crack extension

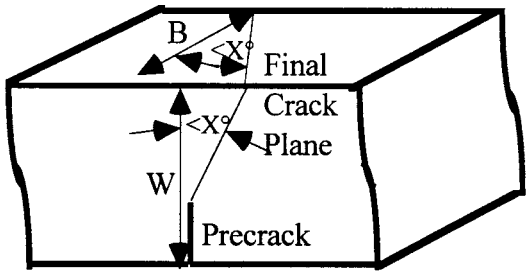


FIG. A2.5 Illustration of Angular Allowance of Final Crack Plane Where X° is 5° for Three-Point Flexure and 10° for Four-Point Flexure

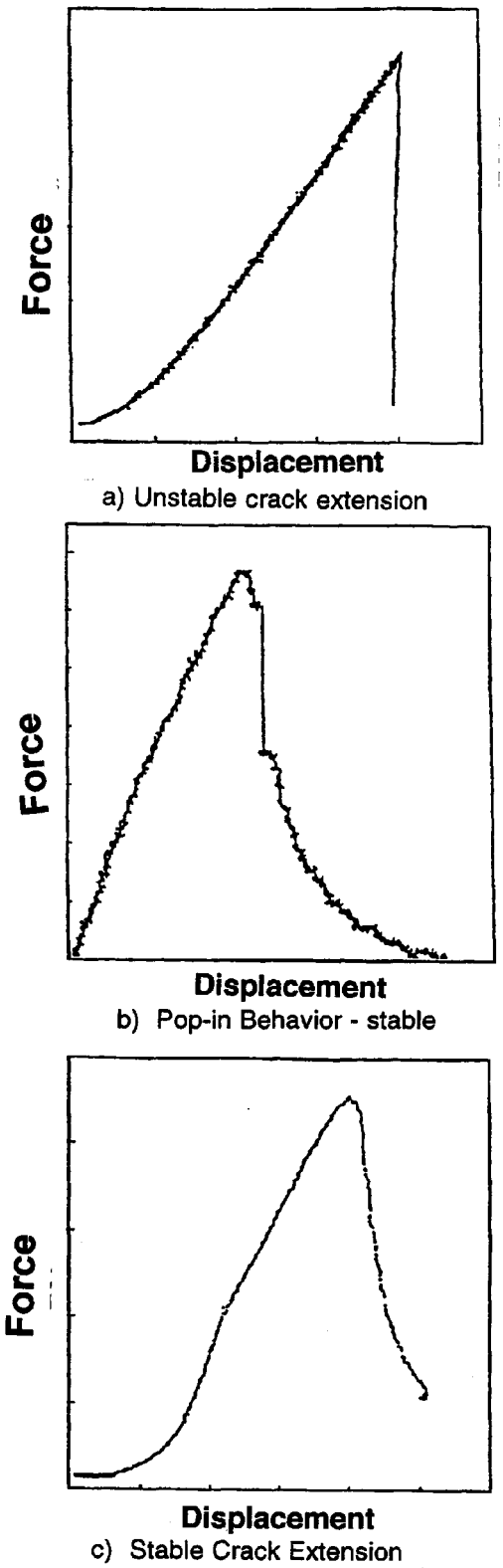


FIG. A2.6 Load Displacement Diagrams from Precracked Beam Tests

may give greater fracture toughness values than those from tests with stable crack extension.

A2.3.8 If there is evidence of environmentally-assisted slow crack growth then it is advisable to run additional tests in an inert environment. Alternatively, additional tests may be done in laboratory ambient conditions at faster or slower test rates than those specified in this standard in order to determine the sensitivity to test rates. Testing rates that differ by two to three orders of magnitude or greater than those specified are recommended. (See 9.3.)

A2.4 Calculation

A2.4.1 Calculate the fracture toughness, K_{Ipb}, for each test specimen and test configuration.

A2.4.2 For three-point flexure with $\frac{S_o}{W} = 4$, $0.35 \leq \frac{a}{W} \leq 0.60$ and a maximum error of 2 % (**19**) :

$$K_{Ipb} = g\left[\frac{P_{max}S_o10^{-6}}{BW^{3/2}}\right]\left[\frac{3[a/W]^{1/2}}{2[1-a/W]^{3/2}}\right] \quad \text{(A2.1)}$$

where:

$$g = g(a/W) = \frac{1.99 - [a/W][1 - a/W][2.15 - 3.93[a/W] + 2.7[a/W]^2]}{1 + 2[a/W]} \quad \text{(A2.2)}$$

Eq A2.1 and Eq A2.2 have also been used for $\frac{S_o}{W} = 5$ (**20**) with maximum errors of 1.5 % for $0.35 \leq \frac{a}{W} \leq 0.60$.

Example—For $W = 4.00$ mm $= 4.00 \times 10^{-3}$ m, $a_o = 2.00$ mm $= 2.00 \times 10^{-3}$ m and
$S_o = 16.0$ mm $= 16.0 \times 10^{-3}$ m then
$a/W = 0.50$, $S_o/W = 4.0$, $g = 0.8875$.

A2.4.3 For three-point flexure with $5 \leq \frac{S_o}{W} \leq 10$, $0.35 \leq \frac{a}{W} \leq 0.60$ and a maximum error of 1.5 % (**9**):

$$K_{Ipb} = g\left[\frac{P_{max}S_o10^{-6}}{BW^{3/2}}\right]\left[\frac{3[a/W]^{1/2}}{2[1-a/W]^{3/2}}\right] \quad \text{(A2.3)}$$

where:

$$g = g(a/W) = A_o + A_1(a/W) + A_2(a/W)^2 + A_3(a/W)^3 + A_4(a/W)^4 + A_5(a/W)^5 \quad \text{(A2.4)}$$

where coefficients for g are shown in Table A2.1

Example—For $W = 4.00$ mm $= 4.00 \times 10^{-3}$ m, $a_o = 2.00$ mm $= 2.00 \times 10^{-3}$ m and
$S_o = 40.0$ mm $= 40.0 \times 10^{-3}$ m then
$a/W = 0.50$, $S_o/W = 10.0$, $g = 0.9166$.

A2.4.4 For four-point flexure with $0.35 \leq \frac{a}{W} \leq 0.60$ and a maximum error of 2 % (**21**):

$$K_{Ipb} = f\left[\frac{P_{max}[S_o - S_1]10^{-6}}{BW^{3/2}}\right]\left[\frac{3[a/W]^{1/2}}{2[1-a/W]^{3/2}}\right] \quad \text{(A2.5)}$$

where:

$$f = f(a/W) = 1.9887 - 1.326[a/W] - \frac{\{3.49 - 0.68[a/W] + 1.35[a/W]^2\}[a/W]\{1 - [a/W]\}}{\{1 + [a/W]\}^2} \quad \text{(A2.6)}$$

Example—For $W = 4.00$ mm $= 4.00 \times 10^{-3}$ m, $a_o = 2.00$ mm $= 2.00 \times 10^{-3}$ m,

TABLE A2.1 Coefficients for the Polynomial *g(a/W)* for Three-point Flexure

	S_o/W				
	5	6	7	8	10
A_o	1.9109	1.9230	1.9322	1.9381	1.9472
A_1	−5.1552	−5.1389	−5.1007	−5.0947	−5.0247
A_2	12.6880	12.6194	12.3621	12.3861	11.8954
A_3	−19.5736	−19.5510	−19.0071	−19.2142	−18.0635
A_4	15.9377	15.9841	15.4677	15.7747	14.5986
A_5	−5.1454	−5.1736	−4.9913	−5.1270	−4.6896

TABLE A2.2 Precracked Beam Results from VAMAS Round Robin for Gas-Pressure Sintered Silicon Nitride (13,22,23)

Test Rates mm/s[A]	Number of Laboratories[B]	Overall Mean MPa√m	Repeatability (Within-Laboratory)			Reproducibility (Between-Laboratories)		
			Std Dev MPa√m	95 %limit MPa√m	COV[C] %	Std Dev MPa√m	95 %limit MPa√m	COV[C] %
0.0166 or (0.0083)	16	5.77	0.26	0.72	4.5	0.51	1.42	8.8
0.000083 or (0.000167, 0.000042)	12	5.60	0.26	0.73	4.7	0.40	1.11	7.1

[A] Numbers in parentheses show alternative test rates that some laboratories used rather than the specified rates.
[B] At each test rate the results from one laboratory were deleted, due to high within-laboratory (repeatability) scatter.
[C] Coefficient of variation.

S_o = 40.0 mm = 40.0 $\times 10^{-3}$ m and S_i = 20.0 mm = 20.0 $\times 10^{-3}$ m then
a/W = 0.50, f = 0.9382.

where:

K_{Ipb} = fracture toughness (MPa $\sqrt{\text{m}}$),
$f = f(a/W)$ = function of the ratio a/W for four-point flexure,
$g = g(a/W)$ = function of the ratio a/W for three-point flexure,
P_{max} = maximum force (N),
S_o = outer span (m),
S_i = inner span (m),
B = side to side dimension of the test specimen perpendicular to the crack length (depth) as shown in A2.4 (m),
W = top to bottom dimension of the test specimen parallel to the crack length (depth) as shown in A2.4 (m), and
a = crack length as determined in A2.4 (m).

A2.5 Valid Test

A2.5.1 A valid pb test shall meet the following requirements in addition to the general requirements of these test methods (9.2):

A2.5.1.1 Test specimen size (A2.1.1) shall be 3 by 4 mm with tolerances as shown in Fig. A2.1 and the length shall be at least 20 mm but not more than 50 mm unless test specimens of larger cross section are used as long as the proportions given in Fig. A2.1 are maintained.

A2.5.1.2 Test specimen preparation (A2.1.2) shall conform to the procedures of A2.1.2.

A2.5.1.3 Crack starter (A2.3.1) introduced from Vickers indent shall be produced at an indent force ≤ 100 N and one of the diagonals of each of the indents shall be aligned parallel to the test specimen length.

A2.5.1.4 Pop-in precrack (A2.2.2 and A2.3.2) shall be introduced using a grooved compression fixture.

A2.5.1.5 Crack length (A2.3.5): difference between average crack length and minimum precrack length shall be less than 10 % and average precrack length shall be $0.35W < a < 0.6W$.

A2.5.1.6 Plane of final crack (A2.3.6) shall be parallel to both the test specimen dimensions B and W within ± 5° for three-point flexure and ± 10° for four-point flexure.

A2.6 Reporting Requirements

A2.6.1 In addition to the general reporting requirements of 10.1, 10.2 and 10.3 report the following for the pb method.

A2.6.1.1 Mean crack length as measured in A2.3.5 (mm),

A2.6.1.2 Each applied force-displacement (time or strain) diagram,

A2.6.1.3 Precracking details, such as the number of indents, indentation force and the force rate during pop-in.

A2.7 Precision and Bias

A2.7.1 *Precision*

The precision of the pb method depends upon the uncertainties in the measured break load, P; the precrack size, a; the quality of the precrack (e.g., evenness, straightness, residual stresses); the beam dimensions, B and W; the stress intensity factor coefficient, Y; the quality and alignment of the precrack, and whether any stable crack extension occurs and whether it is detected and measured.

A2.7.1.1 Results from an eighteen-laboratory, international round robin conducted under the auspices of the Versailles Advanced Materials and Standards (VAMAS) can be used to estimate the precision of the pb method **(13, 22, 23)** . A gas pressure sintered silicon nitride was tested by procedures that were similar to those prescribed in this Test Method. An important exception was that specific actuator displacement (stroke) rates were prescribed, rather than stress intensity factor rates. Two actuator displacement (stroke) rates, 0.0166 mm/s and 0.0000833 mm/s were prescribed. This permitted an assessment of whether time-dependent environmental effects were present. Ten test specimens were tested at each test rate

by each laboratory. A variety of test fixtures and test rates were used for precracking. The results were analyzed in accordance with Practices E177 and E691. The results are given in Table A2.2.

A2.7.1.2 The VAMAS round robin also included pb testing on a zirconia-alumina composite material. Environmentally assisted crack growth and possible rising R-curve behavior caused complications in interpretation of the results as discussed in Ref. (13) .

A2.7.2 *Bias*

The bias is estimated to be negligible (<1%) when using the pb method for a material with a flat R-curve and which has no susceptibility to environmentally-assisted slow crack growth. pb data for SRM 2100 are virtually identical agreement with sc and vb results.

A2.7.3 A slight loss of accuracy and precision may result from the use of very short 3–point spans as discussed in Ref (**12**). The precrack angle and middle-roller fixture alignment tolerances specified in this standard lead to a maximum possible 3 % error in $K_{I,\ pb}$.

A3. PROCEDURES AND SPECIAL REQUIREMENTS FOR THE SURFACE-CRACK IN FLEXURE METHOD

A3.1 Test Specimen

A3.1.1 *Test Specimen Size*—The test specimen shall be 3 X 4 mm in cross section with the tolerances shown in Fig. A3.1. The length shall be 45 to 50 mm. Half length test specimens with cross-section dimensions of 3 X 4 mm and lengths of 25 mm or greater may also be used.

A3.1.2 *Test Specimen Preparation*—Test specimens prepared in accordance with the Procedure of Test Method C1161, test specimen Type B, are suitable as summarized in the A3.1.2.1-A3.1.2.4. Alternative procedures may be utilized provided that unwanted machining damage and residual stresses are minimized. Report any alternative test specimen preparation procedure in the test report.

A3.1.2.1 All grinding shall be done with an ample supply of appropriate filtered coolant to keep workpiece and wheel constantly flooded and particles flushed. Grinding shall be in at least two stages, ranging from coarse to fine rates of material removal. All machining shall be in the surface grinding mode parallel to the test specimen long axis. No Blanchard or rotary grinding shall be used. The stock removal rate shall not exceed 0.02 mm per pass to the last 0.06 mm per face. These conditions are intended to minimize machining damage or surface residual stresses which can strongly affect tests using sc test specimens. As the grinding method of Test Method C1161 is well established and economical, it is recommended.

A3.1.2.2 For all surfaces except that to be indented perform finish grinding with a diamond-grit wheel of 320 grit or finer. No less than 0.06 mm per face shall be removed during the final finishing phase, and at a rate of not more than 0.002 mm per pass.

A3.1.2.3 The indentation can be placed in either a 3- or 4-mm wide flat surface of the beam. The surface need not have an optical quality finish. It need only be flat such that the indentation is not affected by machining striations and marks. For the surface to be indented (either the 3- or 4-mm dimension), a diamond-grit wheel (320 to 500 grit) shall be used to remove the last 0.04 mm at a rate of not more than 0.002 mm per pass. Polish, lap or fine grind this face to provide a flat, smooth surface for the surface crack. It can alternatively be ground with a 600-grit or finer wheel, provided that residual stresses are not introduced.

A3.1.2.4 The two end faces need not be precision machined. The four long edges shall be chamfered at 45° a distance of 0.12 ± 0.03 mm, or alternatively, they may be rounded with a radius of 0.15 ± 0.05 mm as shown in Fig. A3.1. Edge finishing shall be comparable to that applied to the test specimen surfaces. In particular, the direction of the machining shall be parallel to the test specimen long axis.

A3.1.3 It is recommended that at least ten and preferably twenty test specimens be prepared. This will provide test specimens for practice tests to determine the best indentation force. It will also provide make up test specimens for unsuccessful or invalid tests so as to meet the requirements of 9.1 and 9.2.

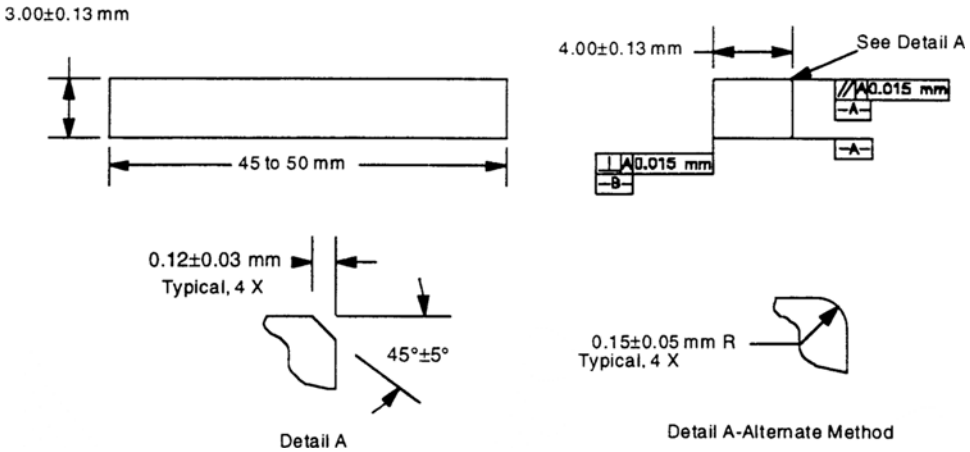


FIG. A3.1 Dimensions of Rectangular Beam

A3.2 Apparatus

A3.2.1 *General*—Conduct this test in four-point flexure. A displacement measurement is not required.

A3.2.2 *Fracture Test Fixture*—The general principles of the four-point test fixture are detailed in 7.4 and illustrated in A1.1.

A3.3 Procedure

A3.3.1 *Precracking—Standard Procedure:*

A3.3.1.1 Use a Knoop indenter to indent the middle of the polished surface of the test specimen. Orient the long axis of the indent at right angles (within 2°) to the long axis of the test specimen as shown in Fig. A3.2. Tilt the test specimen ¼ ° to ½ ° as shown in Fig. A3.3. The 1/4° to 1/2° tilt is intended to make the precrack easier to discern during measurement of precrack size after fracture. Use a full-force dwell time of 15 s or more during the indentation cycle. Indentation times longer than 15 s may be helpful for some materials such as zirconia. A schematic of a resulting precrack is shown in Fig. A3.4 and Fig. A3.3.

A3.3.1.2 The indentation force, *F*, used may have to be determined for each different class of material by the use of a few trial test specimens. The force must be great enough to create a crack that is greater than the naturally-occurring flaws in the material, but not too great relative to the test specimen cross section size, nor so great that extreme impact damage occurs. Indentation forces of approximately 10 to 20 N are suitable for very brittle ceramics, 25 to 50 N for medium "tough" ceramics, and 50 to 100 N for very "tough" ceramics. Indentation loads of 100 N –150 N may be necessary for materials with medium to coarse-grain sizes. In such materials, it is necessary to make large precracks that will stand out against the normal microstructural roughness on the fracture surface. An indentation force of 30 N may be suitable for most glasses. The Knoop indentation procedure to create a surface crack will not be successful on very soft (low hardness) or porous ceramics, since precracks do not form under the indentation. The indentation procedure also may not work on very "tough" ceramics, since they are resistant to the formation of a crack or the crack is so small that it is removed during the subsequent material removal step (A3.3.2) to remove the residual stress and damage zone. A trial specimen may be tested to help determine the best indentation load. Indent then fracture the specimen in the flexure fixtures without removal of the indentation and residual stress damage zone. Examine the fracture surface to confirm that the specimen has fractured from the precrack, that the precrack is discernible, and within the prescribed size limits. Do not use the outcome from such a trial test in the final analyses, however, since the residual indentation stresses have not been removed.

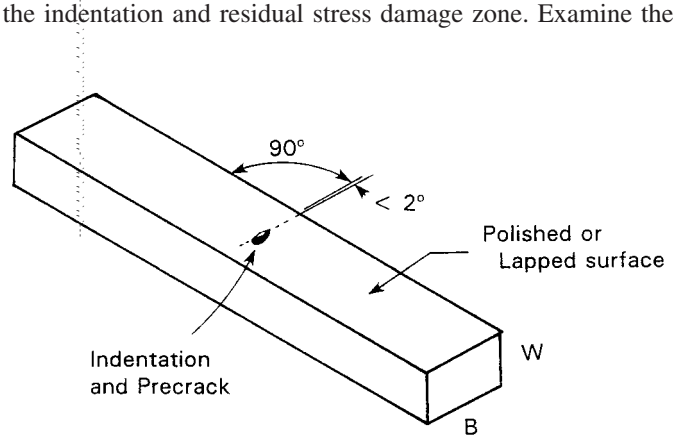


FIG. A3.2 Surface-Crack in Flexure (sc) Test Specimen

A3.3.2 *Removal of Indented Zone:*

A3.3.2.1 Measure the length of the long diagonal, *d*, of the Knoop impression to within 0.005 mm. This measurement need not be done to the precision required for hardness measurements. If Knoop hardness is to be reported, greater care should be exercised in making the diagonal size measurement and in the preparation of the initial test specimen surface.

Calculate the approximate depth, *h*, of the Knoop impression as follows:

$$h = d/30 \quad \text{(A3.1)}$$

A3.3.2.2 Measure the initial (pre-polishing) test specimen dimension, *W*, at the indent location to within 0.002 mm. A hand-held micrometer with a vernier graduation is suitable.

A3.3.2.3 Mark the side of the test specimen with a pencil-drawn arrow in order to indicate the surface with the precrack and its approximate location.

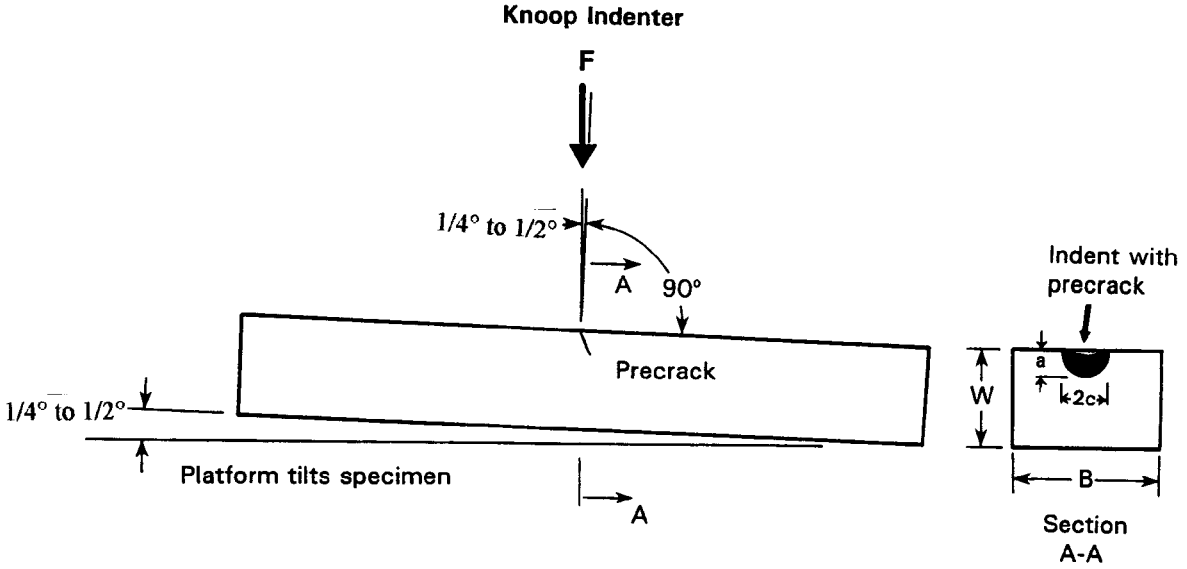
A3.3.2.4 Remove the residual stress damage zone by mild grinding, hand grinding, or hand polishing with abrasive papers.

A3.3.2.5 Hand lapping or grinding may be done wet or dry, with the type of procedure reported. Remove an amount of material that is approximately equal to 4.5 to 5.0 *h* as shown in Fig. A3.5. If there is evidence that this material removal has not eliminated deep lateral cracks, then additional material should be removed as described in A3.3.2.6. The material removal process shall not induce residual stresses or excessive machining damage in the test specimen surface. Remove the last 0.005 mm with a finer grit (220 to 280 grit) paper with less pressure, so as to minimize polishing damage. Check the test specimen dimension, *W*, frequently during this process. In particular, the evenness of *W* should be monitored. A hand micrometer should be used to check *W* at several locations across the specimen width *B* in the vicinity of the indentation. Use a hand micrometer with a resolution of 0.0025 mm or better.

NOTE A3.1—Hand grinding the test specimen with 180 to 220 grit silicon carbide paper can remove the required amount in 1 to 5 min per test specimen for many ceramics. Faster removal rates occur when hand grinding dry. Finer-grit (320 to 400 grit) papers are recommended for glasses for both rough- and fine- grinding steps. Diamond impregnated abrasive disks with 30 μm or finer abrasive may also be used. Hand grinding, hand lapping or hand polishing may not be effective with very hard ceramics. For very hard ceramics, see A3.3.2.7.

NOTE A3.2—Hand lapping or grinding may make the surface uneven or not parallel to the opposite test specimen face. This can cause misalignments during subsequent testing on test fixtures. If the polished face cannot be maintained parallel to the opposite face within ± 0.015 mm, then fully-articulating fixtures should be used for flexure testing in accordance with 7.4.3. A slight rounding of the edges of the test specimen from hand grinding is usually inconsequential. In a given test specimen, regularly change the orientation of the surface being polished to the lapping disk during material removal steps to minimize unevenness.

NOTE A3.3—**Warning:** Fine ceramic powders or fragments may be created if the lapping or hand grinding is done dry. This can create an inhalation hazard if the ceramic contains silica or fine whiskers. Masks or respirators should be used, or the removal should be done wet.



NOTE 1—The indent and precrack sizes are exaggerated for clarity.

FIG. A3.3 The Test Specimen may be Indented at a ½ ° Tilt in Order to Enhance the Chances of Detecting the Precrack on the Fractographic Surface During Subsequent Fracture Analysis. The indentation may be introduced in either the narrow 3-mm face or the wide 4-mm face.

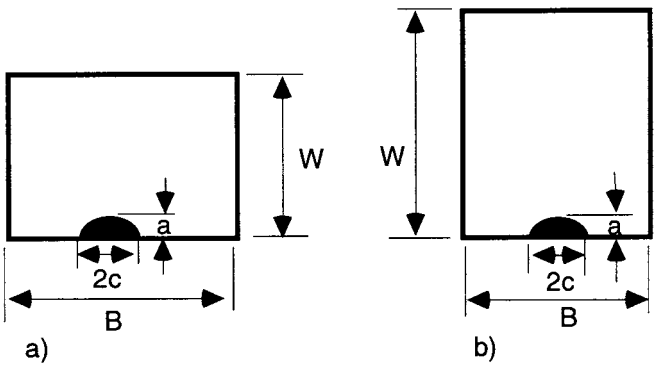
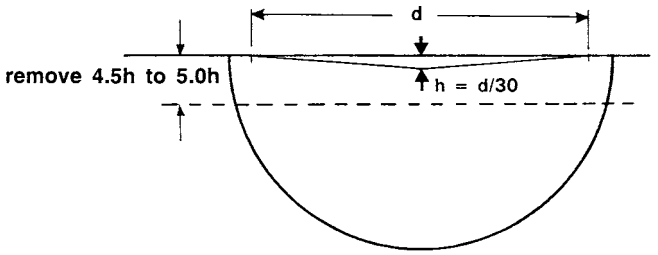


FIG. A3.4 a and b Cross Section of sc Test Specimens Showing the Precrack Configurations for Two Orientations



NOTE 1—Remove 4.5*h* to 5.0*h* from the test specimen surface in order to remove the indent and damage zone.

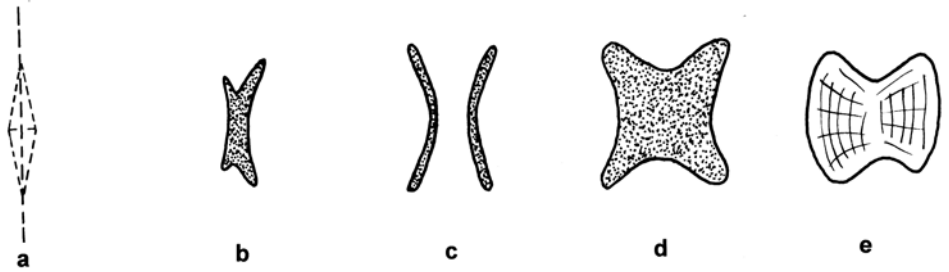
FIG. A3.5 The Precrack Extends Below the Knoop Hardness Impression, which has Depth, *h*

NOTE A3.4—Removing 4.5 to 5.0 *h* eliminates the residual stress damage zone under the impression, and usually leaves a precrack shape that has the highest stress intensity factor at the deepest part of the precrack periphery. The location of the maximum stress intensity can be controlled by the amount of material removed. The initial precrack under the Knoop indent is roughly semicircular and Y_{max} is at the surface. As material is removed, the precrack becomes more semi-elliptical in shape (or like a section of a circle) and Y_{max} shifts to the deepest part of the precrack. If too much material is removed, the remaining precrack will be too small and fracture will not occur from the precrack. In such cases smaller amounts should be removed, provided that no less than 3 *h* is removed. If this step is not adequate to ensure fracture from the precrack, then a greater indent force or the alternative procedure described in Appendix X3 may be used.

A3.3.2.6 After the prescribed amount of material has been removed, examine the ground-indented surface for evidence of remnant lateral cracks. Fig. A3.6 provides guidance. A low power reflected light metallurgical optical microscope with magnifications from ×100 – ×400 may be used to examine the ground-indented tensile surface. If there is evidence of remnants of lateral cracks, then additional material should be removed (6*h* –10*h*) to ensure that the lateral cracks remnants are removed. Remnant lateral cracks are more apt to be a problem with brittle materials (for example, K_{Isc} < 3.0 MPa$\sqrt{}$m) or if larger indentation loads (≥ 98 N) are used.

A3.3.2.7 Surface grinding with diamond wheels is also permitted as a means to remove the indent and residual stress damage zone, but it is much more difficult to ensure that the correct amount of material has been removed from each test specimen. There also is a potential for introduction of residual stresses. Machine grinding will be necessary for very hard ceramics. If machine grinding is used, use fine wheel grits and small removal rates.

A3.3.2.8 If water or a cutting fluid is used, then ensure that the test specimen is dry (for example, by heating) prior to fracture testing. There is no consensus on the best conditions for drying specimens. Heating in an air or vacuum oven at 100 °C – 150 °C for times up to 1 h and then storage in a desiccator prior to testing may be sufficient.



NOTE 1—Remnants of lateral cracks may be visible on the ground tensile surface after removal of the damage zone in some brittle materials. (a) has no trace of lateral cracks. The Knoop indentation (dashed line) has been removed and the median crack is very tight and not visible. (b) through (e) show examples of remnants of lateral cracks.

FIG. A3.6 Remnants of Lateral Cracks

A3.3.2.9 Annealing or heat treating to remove the residual stresses under the indent are not permitted by this standard due to the risk of crack tip blunting, crack healing, or possible changes in the microstructure.

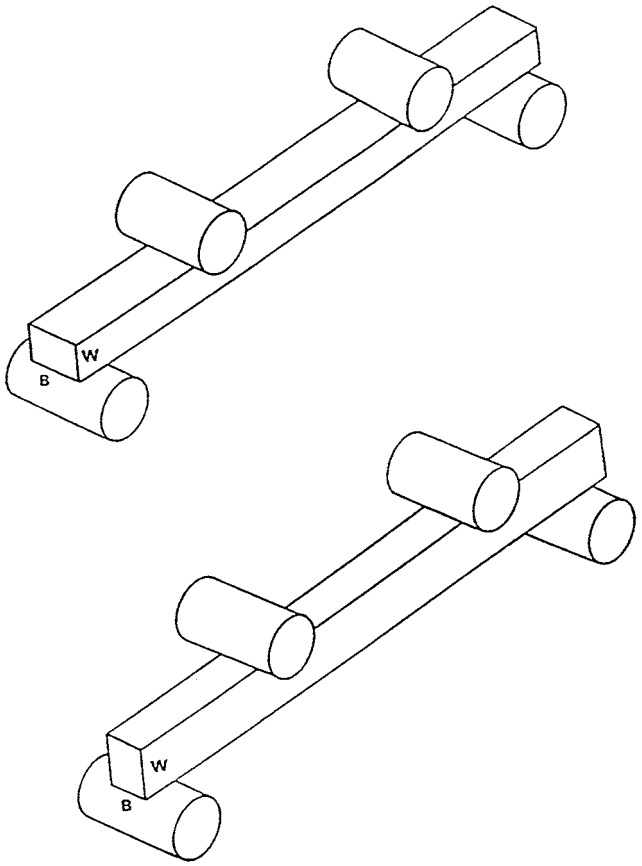
A3.3.2.10 Measure and record the final (post-polishing) test specimen dimensions, *B* and *W*, in the vicinity of the precrack to within 0.002 mm.

A3.3.2.11 In some materials a dye penetrant may aid pre-crack detection after the test specimen is fractured. If a dye penetrant is used, the specimen should be dried thoroughly prior to fracture.

A3.3.3 *Fracture Test*—Insert the test specimen into the test fixture as shown in Fig. A3.7, with the surface crack on the tension face, within 1.0 mm of the midpoint between the two inner rollers, S_i, of the four-point test fixture. Align the test specimen so that it is centered directly below the axis of the force application. Full length test specimens (45 to 50 mm length) should be tested on 20 mm X 40 mm test fixtures and half length test specimens (~25 mm length) should be tested on 10 mm X 20 mm test fixtures. The test specimen may be preloaded to approximately 25 % of the expected fracture force. Place cotton, crumbled tissue, or other appropriate material under the test specimen to prevent the pieces from impacting the fixture upon fracture. Place a thin shield around the fixture to ensure operator safety and to preserve the primary fracture pieces for subsequent fracture analysis. Test the test specimen to fracture at rates in accordance with 9.6.

NOTE A3.5—The force rate will range from 10 to 250 N/s for a test specimen with *B*=4 mm, *W*=3 mm, with a precrack size, a, of 100 μm, on a four-point test fixture with S_o = 40 mm. If the test specimen is tested on edge (*B* = 3 mm, *W* = 4 mm), the rates will be 13 - 388 *N/s*. Rates for alternative geometries and precrack sizes can be estimated from Eq A3.9 with an approximation of *Y* = 1.3. Displacement rates of 0.002 to 0.10 mm/s will be suitable for a 3 by 4-mm test specimen with a 100 μm precrack in the 4-mm (*B*) face.

A3.3.4 *Post Test Measurements*—Examine the fracture surfaces of the test specimen and measure the initial precrack dimensions, *a* and 2*c*, as shown in Fig. A3.4 and Fig. A3.3. Fractographic techniques and fractographic skills are needed for this step (see Appendix X1). If stable crack extension is not detected, the critical crack size should be the same as the precrack size. Measure the crack depth, *a*, to within 0.005 mm (5 μm) or less if possible and the crack width, 2*c*, to within



NOTE 1—The precrack must be on the tension (bottom) surface.

FIG. A3.7 The Flexure Specimen Can be Tested with Either the Wide or Narrow Face on the Flexure Rollers

0.010 mm (10 μm) or less if possible. If stable crack extension is detected, measure both the initial and final crack sizes.

A3.3.4.1 The precision of the crack size measurement depends upon the material and its microstructure, the clarity of the crack, and the mode of viewing. In many instances, the computed fracture toughness is not very sensitive to the precision of the crack size measurement as discussed in References 14 and 24. Depending upon the crack sizes and specimen geometries, satisfactory estimates of fracture toughness may be obtained even with crack size measurements that are less precise than suggested above. The optimum procedure

will vary from material to material. Either an optical microscope or a scanning electron microscope can be used. Low magnifications (×50-100) can be used to locate the pre-crack, and intermediate magnifications (×100-500) to photograph the pre-crack for measurement. If an optical microscope is used, then variation of the lighting source and direction can be used to highlight the pre-crack. A stage micrometer shall be used to confirm the magnifications. If a scanning electron microscope is used, then it is recommended that a SEM magnification calibration standard be used to confirm the magnification. In some instances dye penetrants may be useful, but care should be taken to ensure that the dyes are completely dry during the fracture test to preclude undesired slow crack growth or undesired crack face bonding.

A3.3.4.2 Additional details on techniques to find and characterize the pre-cracks are given in Appendix X1 and Appendix X2 and Ref (**24**).

A3.3.5 Measure the cross section dimensions B and W of each test specimen to within 0.002 mm.

A3.4 Calculation

A3.4.1 Calculate the stress intensity shape factor coefficients for both the deepest point of the precrack periphery, Y_d, and for the point at the surface, Y_s which will give a maximum error of 3 % for an "ideal" precrack and an estimated maximum error of 5 % for a "realistic" precrack.

NOTE A3.6—The stress intensity factor coefficients are from Newman and Raju, Ref (**25**), and are the same as those used in Practice E740. These coefficients are valid only for a/c ≤ 1. They can be used for a/c ratios slightly greater than 1 with a slight loss of accuracy.

A3.4.1.1 For the deepest point of the precrack:

$$Y_d = \frac{[\sqrt{\pi} M H_2]}{\sqrt{Q}} \tag{A3.2}$$

where:

$$Q = Q(a/c) = 1 + 1.464[a/c]^{1.65} \tag{A3.3}$$

$$\begin{aligned} M &= M(a/c, a/W) \\ &= [1.13 - 0.09[a/c]] + \left[-0.54 + \frac{0.89}{[0.2 + [a/c]]}\right][a/W]^2 \\ &\quad + \left[0.5 - \frac{1}{[0.65 + [a/c]]} + 14[1 - a/c]^{24}\right][a/W]^4 \end{aligned} \tag{A3.4}$$

$$\begin{aligned} H_2 &= H_2(a/c, a/W) = 1 - [1.22 + 0.12[a/c]]\,[a/W] \\ &\quad + [0.55 - 1.05[a/c]^{0.75} + 0.47[a/c]^{1.5}]\,[a/W]^2 \end{aligned} \tag{A3.5}$$

A3.4.1.2 For the point at the surface:

$$Y_s = \frac{[\sqrt{\pi} M H_1 S]}{\sqrt{Q}} \tag{A3.6}$$

where:

$$H_1 = H_1(a/c, a/W) = 1 - [0.34 + 0.11[a/c]]\,[a/W] \tag{A3.7}$$

$$S = S(a/c, a/W) = [1.1 + 0.35[a/W]^2]\sqrt{a/c} \tag{A3.8}$$

Example—For W=3× 10^{-3} m, a=50×10^{-6} m and $2c$=120×10^{-6} m
a/c=0.833, a/W=0.017, Y_d=1.267 and Y_s=1.292

A3.4.1.3 If the test specimens are chamfered, and if the chamfer sizes are larger than 0.15 mm, then the fracture toughness values should be corrected in accordance with Appendix X4.

A3.4.2 Use the greater value of Y_d or Y_s for Y and then calculate the fracture toughness, K_{Isc}, from the following equation:

$$K_{Isc} = Y\left[\frac{3P_{\max}[S_o - S_i]10^{-6}}{2BW^2}\right]\sqrt{a} \tag{A3.9}$$

where:

K_{Isc} = the fracture toughness (MPa $\sqrt{\text{m}}$),
Y = the stress intensity factor coefficient (dimensionless),
$P_{\max}$ = the maximum force (N),
S_o = the outer span (m),
S_i = the inner span (m),
B = the side to side dimension of the test specimen perpendicular to the crack length (depth) as shown in Fig. A3.2, Fig. A3.3 and Fig. A3.4 (m),
W = the top to bottom dimension of the test specimen parallel to the crack length (depth) as shown in Fig. A3.2, Fig. A3.3 and Fig. A3.4 (m),
a = the crack depth (m) as shown in Fig. A3.3 and Fig. A3.4 and
c = the crack half width (m) as shown in Fig. A3.3 and Fig. A3.4.

NOTE A3.7—The term in brackets in Eq A3.9 is the flexural strength (in MPa) of the beam with a surface crack. It is often useful to compare this value with the range of values of the flexural strength of test specimens without a precrack, in which fracture occurs from the natural fracture sources in the material.

A3.5 Requirements

A3.5.1 The use of the semi-ellipse to model the precrack shape is an approximation which is most valid for instances where the greatest stress intensity factor coefficient is at the deepest part of the precrack (Y_{max}= Y_d). If the maximum stress intensity factor coefficient is at the surface (Y_{max}= Y_s), then the semi-ellipse may not necessarily be an adequate model of the precrack. In such a case, re-examine the precrack shape. If the precrack is not semi-elliptical, reject the datum.

A3.5.2 If the precrack form is severely distorted in the third dimension (i.e. is not flat), or the form of the precrack is incomplete over more than 33 % of its periphery, reject the datum.

A3.5.3 If hand grinding or machining damage (see A3.3.2) interfere with the determination of the precrack shape and Y_s is greater than Y_d, then reject the datum.

A3.5.4 If the precrack shows evidence of excessive extension (corner pop-in) at the intersection of the surface, then reject the datum (see example in X2.1)

A3.5.5 If the precrack shows evidence of stable extension prior to instability, then measure both the initial precrack size, and the critical crack size. Stable crack extension may manifest itself as a halo around the precrack. See examples in X2.1 and Reference (35) for additional information. Report both the apparent fracture toughness using the initial precrack size, K_{Isc}, and the apparent fracture toughness at instability, K_{Isc}*.

NOTE A3.8—It has been common practice to calculate a nominal fracture toughness value based on the maximum force and the original crack dimensions before testing for use as an aid in interpreting sc test results. If significant stable crack growth occurs, the original crack dimensions may no longer be pertinent. If stable extension is due to environmentally-assisted slow crack growth, the nominal fracture toughness will underestimate K_{Isc} in the absence of environmental effects. Alternatively, if the stable crack extension is due to rising R-curve behavior, the calculated fracture toughness using the initial precrack size will underestimate the fracture toughness at criticality.

A3.5.6 If there is evidence of environmentally-assisted slow crack growth then it is advisable to run additional tests in an inert environment. Alternatively, additional tests may be done in laboratory ambient conditions at faster or slower test rates than those specified in this standard in order to determine the sensitivity to test rates. Testing rates that differ by two to three orders of magnitude or greater than those specified are recommended. (See 9.3.)

A3.6 Valid Test

A3.6.1 A valid sc test shall meet the following requirements in addition to the general requirements of this standard (9.2):

A3.6.1.1 Test specimen size (A3.1.1) shall be 3 by 4 mm with tolerances as shown in Fig. A3.1 and the length shall be 45 to 50 mm.

A3.6.1.2 Test specimen preparation (A3.1.2) shall conform to the procedures in A3.1.2.

A3.6.1.3 Precrack (A3.3.1) introduced from a Knoop indent or the alternative procedure with canted Vickers indent (Appendix X3) shall be produced in the middle of the polished surface with the long axis of the indent at right angles to the long axis of the test specimen (A3.3.1.1), shall be semi-elliptical (A3.5.1), shall not be severely distorted or incomplete (A3.5.2), shall not have been affected by removal of the residual stress field and shall not have Y_s greater than Y_d (A3.5.3) and shall not show evidence of excessive extension (corner pop-in) at the intersection of the surface (A3.5.4).

A3.6.1.4 Residual stresses associated with the indentation shall be removed in accordance with A3.3.2. Material removal shall not introduce residual stresses or excessive machining damage in the test specimen surface.

A3.7 Reporting Requirements

A3.7.1 In addition to the general reporting requirements of 10.1, 10.2, and 10.3, report the following for the sc method:

A3.7.1.1 If the maximum for Y occurred at the test specimen surface (Y_s) or at maximum crack depth (Y_d),

A3.7.1.2 The precrack indent force, F,

A3.7.1.3 If there is evidence for stable crack extension, then state such in the report and report both K_{Isc}* and K_{Isc} (A3.5.5),

A3.7.1.4 The fractographic equipment (optical or SEM) used to observe and measure the precrack, fractographic observations, and a photograph of a representative sc precrack, and

A3.7.1.5 The average indentation diagonal length, the procedure used to remove the indentation and residual stress zones, and the depth of material removed.

A3.8 Precision and Bias

A3.8.1 *Precision*—The precision of the sc method depends upon uncertainties in the measured break load, P; the measured crack dimensions, a and $2c$; the stress intensity factor coefficient, Y; the specimens dimensions W and B; the flexural fixture spans and alignment; chamfer size corrections; the quality of the precrack (e.g., shape uniformity, planarity, lack of interference from residual stresses or lateral cracks); and whether any stable crack extension occurs and whether it is detected and measured. The precision depends primarily upon the quality of the pre-crack and the accuracy and precision of measurement of the pre-crack size. The flexure strength (which combined the uncertainties of P, fixture alignment errors, and B and W, and the chamfer sizes) is estimated to be accurate and precise to within 2 to 3 %. The stress intensity factors coefficients Y for the pre-cracks are expected to be within 3 to 5 % for the instances where fracture initiates at the deepest point of the pre-crack periphery. Pre-crack sizes can be measured to within 5 % with either optical or electron microscopy provided that the material is conducive to fractographic interpretation. Uncertainties in pre-crack size, a and $2c$, are partially ameliorated by an offsetting influence of the stress intensity factor coefficient, Y, as discussed in detail in Refs **(14)** and **(26)**. In summary, for a material that fractures from the deepest part of the pre-crack, and which has a clearly visible, well-shaped pre-crack, the overall precision of the sc method is approximately ± 5 %.

A3.8.1.1 Results from a twenty-laboratory round robin organized under the auspices of the VAMAS project can be found in Ref (14). Three ceramics were tested with five replicate tests specified per condition and material. The VAMAS round robin

TABLE A3.1 Surface Crack in Flexure Results from VAMAS Round Robin (14)

Material	Number of Laboratories	Total Number of Test Specimens	Overall Mean MPa√m[A]	Overall Std Dev MPa√m[A]	Repeatability (Within-Laboratory)			Reproducibility (Between-Laboratories)		
					Std Dev MPa√m	95 %limit MPa√m	COV %[B]	Std Dev MPa√m	95 %limit MPa√m	COV %[B]
Hot-pressed silicon nitride[C]	19	102	4.56	0.32	0.24	0.68	5.4	0.31	0.86	6.8
Hot-isopressed silicon nitride[C]	15	100	5.00	0.48	0.38	1.07	7.7	0.45	1.25	8.9
Yttria-stabilized zirconia[CD]	7	29	4.47	0.31	0.29	0.83	6.6	0.29	0.83	6.6

[A] Average and standard deviation of all individual test results combined.
[B] Coefficient of variation.
[C] A data set from a single outlier laboratory set was excluded and accounts for a small difference in the numbers quoted in A3.8.2.
[D] The modified indentation method of Appendix X3 was used.

results were analyzed in accordance with Practices E177 and E691 to evaluate precision. The results are given in Table A3.1. The within-laboratory precision estimates ranged from 5.4 % to 7.7 %. The between-laboratory uncertainties were slightly greater.

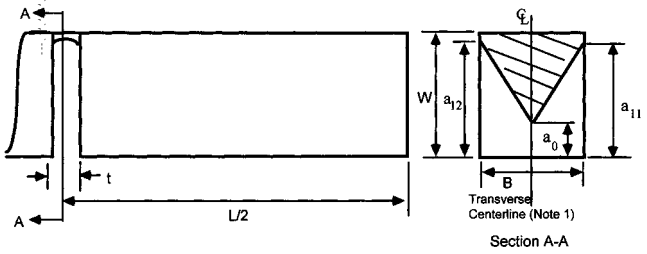
A3.8.2 *Bias*—The bias is estimated to be negligible (< 1 %) when using the sc method with a material with a flat R-curve and which has no susceptibility to environmentally-assisted slow crack growth such as SRM 2100. For example, sc data for SRM 2100 are in nearly identical agreement with pb and vb results. In addition, the grand average of over 100 sc results from 21 laboratories in an international (VAMAS) round robin cited in A3.8.1.1 concurred with the SRM 2100 certified values to within 1 %.

A4. PROCEDURES AND SPECIAL REQUIREMENTS FOR THE CHEVRON NOTCH FLEXURE METHOD

A4.1 Test Specimen

A4.1.1 *Test Specimen Size*—The test specimen has four acceptable geometries as listed in Fig. A4.1 and as shown in Fig. A4.2. Because no generalized, parametric error and sensitivity analysis studies have been conducted on chevron-notched test specimen geometries, this test method focuses on established geometries which reflect a base of experience (that is, those geometries that have been successfully used, studied, and applied under a range of conditions to a variety of materials).

A4.1.2 *Test Specimen Preparation*—Test specimens prepared in accordance with the Procedure of Test Method C1161 are suitable as summarized in A4.1.2.1-A4.1.2.3. Any alternative procedure that is deemed more efficient may be utilized provided that unwanted machining damage and residual stresses are minimized. Report any alternative test specimen preparation procedure in the test report.



Configuration and test fixture	L (mm)	B (mm)	W (mm)	a_0 (mm)	a_{11} and a_{12}	t (mm)
A (Four-point)	45 (min)	3.00±0.13	4.00±0.13	0.80±0.07	0.95W to 1.00W (no overcut)	≤0.25
B (Three-point)	45 (min)	6.35±0.13	6.35±0.13	2.54±0.07	0.95W to 1.00W (no overcut)	≤0.25
C (Four-point)	45 (min)	3.00±0.13	6.00±0.13	1.20±0.07	4.20±0.07 mm	≤0.25
D (Four-point)	45 (min)	3.00±0.13	4.00±0.13	1.40±0.07	0.95W to 1.00W (no overcut)	≤0.25

Note 1-Tip of chevron on transverse centerline shall be within 0.02B.
Note 2-Lengths a_{11} and a_{12} shall be within 0.02W. No overcut of the notch into the topside of the specimen is allowed.
Note 3-Planes from either side of beam which form the chevron shall meet within 0.3t
Note 4-Allowable ranges for a_{11} and a_{12} are in terms of W for Configurations A, B and D and but are given in mm for Configuration C.

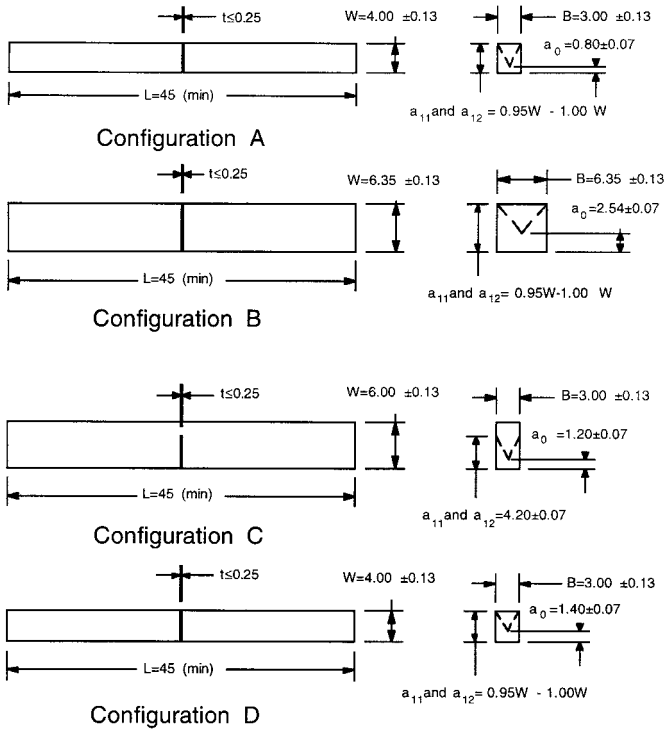
NOTE 1—Tip of chevron on transverse centerline shall be within 0.02*B*.

NOTE 2—Lengths a_{11} and a_{12} shall be within 0.02*W*. No overcut of the notch into the topside of the test specimen is allowed.

NOTE 3—Planes from either side of beam which form the chevron shall meet within 0.3*t*.

NOTE 4—Allowable ranges for a_{11} and a_{12} are in terms of W for Configurations A, B and D and but are given in mm for Configuration C.

FIG. A4.1 Chevron Notch Flexure (vb) Test Specimen Standard Proportions and Tolerances



NOTE 1—All dimensions in mm.

NOTE 2—Tips of chevrons on transverse centerline within 0.02 *B*.

NOTE 3—Planes on either side which form chevrons shall meet within 0.3*t*.

FIG. A4.2 Illustrations of Chevron Notch Flexure (vb) Test Specimen Geometries

A4.1.2.1 All grinding shall be done with an ample supply of appropriate filtered coolant to keep workpiece and wheel constantly flooded and particles flushed. Grinding shall be in at least two stages, ranging from coarse to fine rates of material removal. All machining shall be in the surface grinding mode parallel to the test specimen long axis. No Blanchard or rotary grinding shall be used. The stock removal rate shall not exceed 0.02 mm per pass to the last 0.06 mm per face. These conditions are intended to minimize machining damage or surface residual stresses which can interfere with tests. As the grinding method of Test Method C1161 is well established and economical, it is recommended.

A4.1.2.2 Perform finish grinding with a diamond-grit wheel of 320 grit or finer. No less than 0.06 mm per face shall be removed during the final finishing phase, and at a rate of not more than 0.002 mm per pass.

A4.1.2.3 The two end faces need not be precision machined. No edge treatment (that is, chamfering) of longitudinal edges is allowed on the compression face.

A4.1.3 *Chevron Notch*—Cut the chevron notch using a 180-320 diamond-grit wheel at a rate of not more than 0.002 mm per pass for the final 0.06 mm. The notch thickness, t, should be slightly V-shaped and should be less than 0.30 mm at any point of its intersection with the surface and should be less than 0.150 mm at the root radius of the chevron. (See also requirements in Fig. A4.1 and Fig. A4.2). Planes of notches cut from each side of the test specimen shall meet within 0.3 t. The tip of the chevron shall be on center within 0.02 B. Special machining fixtures for producing chevron notches have been shown to reduce machining costs while increasing the incidence of consistent chevron notches (27). Larger notch thicknesses are acceptable provided that stable crack extension occurs. A V-shaped notch (larger notch width where it intersects the test specimen surface than at the root of the notch) rather than a straight notch shape has resulted in more consistent results (23)

NOTE A4.1—Because no generalized, parametric error and sensitivity analysis studies have been conducted on chevron notch geometries, the notch tolerances given represent those commonly achieved under commercial machining conditions on chevron-notched test specimens which were ultimately used in valid fracture tests **(31)**.

A4.1.4 Prepare at least ten test specimens. This will provide extra test specimens to determine if stable crack growth can be attained without extra preparation (A4.4.1).

A4.2 Apparatus

A4.2.1 *General*—This test is conducted in three-point flexure for one specimen type (geometry B) and four-point flexure for three other specimen types (geometries A, C, D). A displacement measurement (or estimate of displacement from a time sweep) is required.

A4.2.2 *Fracture Test Fixture*—The general principles of three- and four-point test fixtures are detailed in 7.4 and illustrated in Fig. A1.1 and Fig. A1.2, respectively. For four-point flexure the outer and inner spans are S_o = 40 mm and S_i = 20 mm, respectively.

A4.2.2.1 For three-point flexure the support span is S_o = 38-40 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.

A4.2.2.2 For four-point flexure, 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.

A4.3 Procedure

A4.3.1 *Test Specimen Measurement and Alignment*—Measure the cross section dimensions B and W to within 0.002 mm. Measure the notch dimension, a_o, from the chevron tip to the test specimen surface at the notch mouth (that is, opposite the tip of the chevron). Measure the notch dimensions, a_{11} and a_{12}, where the notch groove meets the test specimen surface and calculate a_1, the average of the two values. The difference between the average and the individual values shall be no more than 0.02 W. 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. Orient the chevron tip toward the outer span (that is, the tip of the chevron section is toward the tensile surface). Align the chevron notch with the centerline of the middle roller in the three-point flexure fixture within 0.5 mm or within 1.0 mm of the midpoint between the two inner rollers, S_i, of the four-point flexure fixture. Seat the displacement indicator close to the crack plane if used. Alternatively, use actuator (or crosshead) displacement, back-face strain, or a time sweep.

A4.3.2 *Test Record*—Select a combination of load-sensing device and recording device such that the forces can be obtained from the test record within an accuracy of 1 %. Either load-point displacement, actuator displacement (stroke), displacement of the test specimen at the notch plane, back-face strain or time can be used.

NOTE A4.2—For autographic recording devices choose the sensitivities of force (y-axis) and displacement or time (x-axis) to produce an initial elastic loading trace with a slope between 0.7 and 1.5 (ideally a slope of 1.0) so as to provide a good indication of stable crack growth.

A4.3.3 *Test Rate*—Test the test specimen to fracture at actuator displacement (stroke) rates between 0.0003 to 0.005 mm/s for all the configurations.

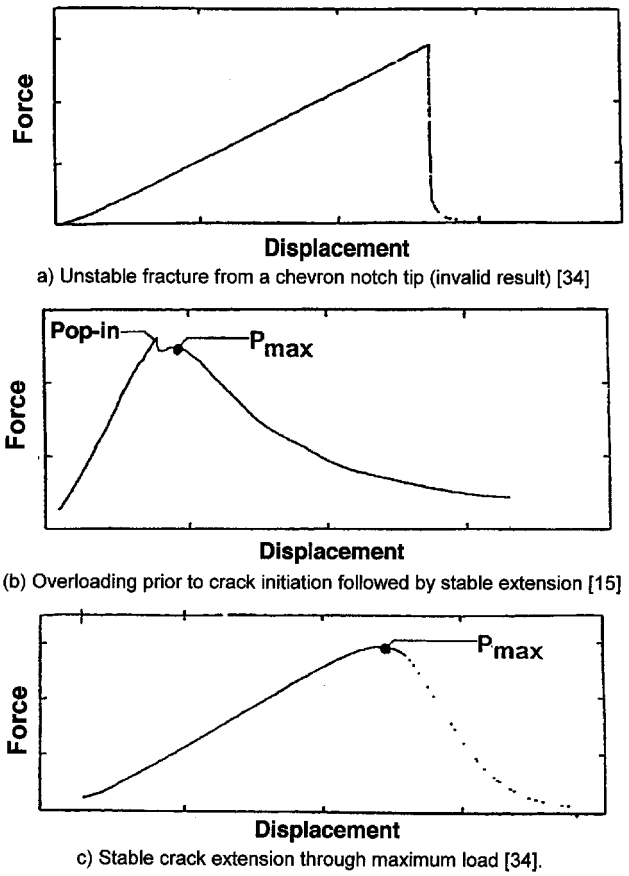
A4.3.4 *Post Test Measurements*—Examine the chevron notch at sufficient magnification (~30×). The tip of the chevron shall be on center within 0.02 B, and the centerline of the notch grooves on either side of the tip shall meet within 0.3 t.

A4.3.5 Examine the fracture surface to determine how well the crack followed the chevron notch plane and separated the test specimen into two pieces. If the "crack follow" through the chevron section was poor, the crack will have deviated substantially farther into one half than the other. If the actual crack surface deviates severely from the intended crack plane as defined by the chevron notch plane, then the test may be invalid.

NOTE A4.3—Deviation of the crack from the notch plane can result from one or more of the following:
(*a*) Strong anisotropy, in which the fracture toughness in the intended crack plane is substantially larger than the fracture toughness in another crack orientation.
(*b*) Coarse-grained or heterogeneous materials.
(*c*) Misalignment of the test specimen in the fixture or an out-of-specification notch.

A4.3.6 *Post Test Interpretation*—The test record shall exhibit a smooth (nonlinear) transition through the maximum force prior to final fracture. If the test specimen exhibits a sudden drop in force from the initial linear portion for the test record not followed by a subsequent force increase, the test is unstable and invalid (See Fig. A4.3a). Determine the relevant maximum test force, P_{max}, from the test record. In some cases the test specimen will overload slightly at crack initiation, as shown in Fig. A4.3b. In the calculations, use the maximum stable force marked P_{max} in Fig. A4.3b and Fig. A4.3c.

A4.3.6.1 If there is evidence of environmentally-assisted slow crack growth then it is advisable to run additional tests in an inert environment. Alternatively, additional tests may be done in laboratory ambient conditions at faster or slower test



a) Unstable fracture from a chevron notch tip (invalid result) [34]

(b) Overloading prior to crack initiation followed by stable extension [15]

c) Stable crack extension through maximum load [34].

FIG. A4.3 Illustrative Applied Force-Displacement Curves: (a) Unstable Fracture from Chevron Tip (34) (Invalid), (b) Overloading Prior to Crack Initiation Followed by Stable Extension (15) and (c) Stable Crack Extension Through Maximum Force (34)

rates than those specified in this standard in order to determine the sensitivity to test rates. Testing rates that differ by two to three orders of magnitude or greater than those specified are recommended. (See 9.3.) However, at actuator displacement rates greater than 0.008 mm/s, stability may be difficult to detect. A combination of testing at fast test rates in inert environment may be compared to laboratory ambient conditions at slow test rates.

A4.4 Recommendations

A4.4.1 In some instances a stable crack will not initiate from the tip of the chevron, resulting in test specimen overload (that is, a force greater than that to produce stable fracture) or underload (that is, a force less than that to produce stable fracture) and catastrophic fracture from the chevron tip, Fig. A4.3a. If this occurs, a simple compression-compression fatiguing procedure to damage the chevron tip, thereby promoting stable initiation and growth of a crack, can be used. The test specimen is placed in the test fixture upside down and the crack tip loaded in compression, several times, to approximately three times the estimated fracture force expected for the normal position. On unloading, remove the test specimen and test it as specified in A4.3.

A4.4.2 Machining of the chevron notch can influence the scatter in the results. Thinner, or more precise notch thicknesses seem to decrease scatter and initiate stable crack growth more readily (**15, 28, 29, 30**). The notch thickness, t, should be in accordance with A4.1.3.

A4.4.3 Actuator displacement (stroke) 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**). In very stiff materials, use of back-face strain is recommended for detection of stable fracture.

A4.5 Calculation

A4.5.1 Calculate the fracture toughness, K_{Ivb}, from the following equation:

$$K_{Ivb} = Y^*_{\min}\left[\frac{P_{\max}[S_o - S_i]10^{-6}}{BW^{3/2}}\right] \quad \text{(A4.1)}$$

where:

K_{Ivb} = the fracture toughness (MPa $\sqrt{\text{m}}$),

$Y^*_{\min}=Y^*_{\min}(a_o/W, a_1/W)$ = the minimum stress intensity factor coefficient as determined from Eq A4.2, Eq A4.3, Eq A4.4 and Eq A4.5 for test specimen geometries A, B, C, and D, respectively (dimensionless),

$P_{\max}$ = the relevant maximum force as determined in A4.3.6 and Fig. A4.3 (N),

S_o = the outer span (m),

S_i = the inner span (m),

B = the side to side dimension of the test specimen perpendicular to the crack length (depth) as shown in Fig. A4.1 and Fig. A4.2 (m),

W = the top to bottom dimension of the test specimen parallel to the crack length (depth) as shown in Fig. A4.1 and Fig. A4.2 (m).

A4.5.1.1 The stress intensity factor coefficient, $Y^*_{\min}$, for geometry A and four-point flexure as derived using a straight through crack assumption and a subsequent curve fit of its relation to a_0/W and a_1/W (**31, 32**) is given as:

$$Y^*_{\min} = \quad \text{(A4.2)}$$

$$Y^*_{\min}(a_o/W, a_1/W) =$$

$$\frac{0.3874 - 3.0919(a_o/W) + 4.2017(a_1/W) - 2.3127(a_1/W)^2 + 0.6379(a_1/W)^3}{1.0000 - 2.9686(a_o/W) + 3.5056(a_o/W)^2 - 2.1374(a_o/W)^3 + 0.0130(a_1/W)}$$

for $0.177 \le a_o/W \le 0.225$ and $0.950 \le a_1/W < 1.000$ and a maximum error of 1 %.

Example—For $W = 4.00$ mm $= 4.00 \times 10^{-3}$ m, $a_o = 0.80$ mm $= 0.80 \times 10^{-3}$ m and
$a_1 = 4.00$ mm $= 4.00 \times 10^{-3}$ m then
$a_o/W = 0.20$, $a_1/W = 1.00$, $Y^*_{\min} = 4.23$.

A4.5.1.2 The stress intensity factor coefficient, $Y^*_{\min}$, for geometry B and three-point flexure as derived using straight

through crack assumption and a subsequent curve fit of its relation to a_0/W and a_1/W (**31, 32**) is given as:

$$Y^*_{min} = \quad \text{(A4.3)}$$

$$Y^*_{min}(a_o/W, a_1/W) =$$

$$\frac{0.7601 - 3.6364(a_o/W) + 3.1165(a_1/W) - 1.2782(a_1/W)^2 + 0.3609(a_1/W)^3}{1.0000 - 3.1199(a_o/W) + 3.0558(a_o/W)^2 - 1.0390(a_o/W)^3 + 0.0608(a_1/W)}$$

for $0.382 \leq a_o/W \leq 0.420$ and $0.950 \leq a_1/W < 1.00$ and a maximum error of 1 %

Example—For W = 6.35 mm = 6.35 $\times 10^{-3}$ m, a_o= 2.54 mm = 2.54$\times 10^{-3}$ m and
a_1= 6.35 mm = 6.35 $\times 10^{-3}$ m then
a_o/W = 0.40, a_1/W=1.00, Y^*_{min}=6.40.

A4.5.1.3 The stress intensity factor coefficient, Y^*_{min}, for geometry C and four-point flexure as derived using Bluhm's slice model and a subsequent curve fit of its relation to a_o/W and a_1/W (**31, 32, 33**) is given as:

$$Y^*_{min} = \quad \text{(A4.4)}$$

$$Y^*_{min}(a_o/W, a_1/W) =$$

$$\frac{1.4680 + 5.5164(a_o/W) - 5.2737(a_1/W) + 8.4498(a_1/W)^2 - 7.9341(a_1/W)^3}{1.0000 + 3.2755(a_o/W) - 4.3183(a_o/W)^2 + 2.0932(a_o/W)^3 - 1.9892(a_1/W)}$$

for $0.184 \leq a_o/W \leq 0.216$ and $0.674 \leq a_1/W \leq 0.727$ and a maximum error of 1 %

Example—For W = 6.00 mm = 6.00 $\times 10^{-3}$ m, a_o= 1.20 mm = 1.20$\times 10^{-3}$ m and
a_1= 4.20 mm = 4.20 $\times 10^{-3}$ m then
a_o/W = 0.20, a_1/W=0.70, Y^*_{min}=2.80.

A4.5.1.4 The stress intensity factor coefficient, Y^*_{min}, for geometry D and four-point flexure as derived using a straight through crack assumption and a subsequent curve fit of its relation to a_0/W and a_1/W (**31, 32**) is given as:

$$Y^*_{min} = \quad \text{(A4.5)}$$

$$Y^*_{min}(a_o/W, a_1/W) =$$

$$\frac{0.5256 - 3.4872(a_o/W) + 3.9861(a_1/W) - 2.0038(a_1/W)^2 + 0.5489(a_1/W)^3}{1.0000 - 2.9050(a_o/W) + 2.7174(a_o/W)^2 - 0.8963(a_o/W)^3 + 0.0361(a_1/W)}$$

for $0.322 \leq a_o/W \leq 0.380$ and $0.950 \leq a_1/W < 1.000$ and a maximum error of 1 %.

Example—For W = 4.00 mm = 4.00 $\times 10^{-3}$ m, a_o= 1.40 mm = 1.40$\times 10^{-3}$ m and
a_1= 4.00 mm = 4.00 $\times 10^{-3}$ m then
a_o/W = 0.35, a_1/W=1.00, Y^*_{min}=5.85.

A4.6 Valid Test

A4.6.1 A valid vb test shall meet the following requirements in addition to the general requirements of these test methods (9.2):

A4.6.1.1 Test specimen size (A4.1.1) shall be as listed in Fig. A4.1 and as shown in Fig. A4.2.

A4.6.1.2 Test specimen preparation (A4.1.2) shall conform to the procedures in A4.1.2.

A4.6.1.3 Chevron notch (A4.1.3 and A4.3.4) shall have planes which meet within 0.3 t, the tip of chevron on the transverse centerline shall be within 0.02 B, and the difference between the average of a_{11} and a_{12}(that is, a_1) and a_{11} or a_{12}, or both, shall not be more than 0.02 W.

A4.6.1.4 Test record (applied force-displacement/time curve) (A4.3.6) shall exhibit smooth (nonlinear) transition through the maximum force prior to final fracture which is indicative of stable crack extension.

A4.7 Reporting Requirements

A4.7.1 In addition to the general reporting requirements of 10.1, 10.2 and 10.3, report the following for the vb method.

A4.7.2 Each flexure diagram with a statement about stability (A4.3.6).

A4.7.3 Include statements about the validity of the chevron notch (A4.3.4) and the crack plane (A4.3.5).

A4.8 Precision and Bias

A4.8.1 *Precision*

The precision of the vb method depends upon the uncertainties in the measured maximum load, P; the quality and geometry of the chevron notch, the beam dimensions, B and W; the stress intensity factor coefficient, Y^*_{min}; and the proper attainment of stable crack extension. No round robin results are available for the vb procedures in this method.

A4.8.2 *Bias*

The bias is estimated to be negligible (< 1 %) when using the vb method with a material with a flat R-curve and which has no susceptibility to environmentally-assisted slow crack growth. vb data for SRM 2100 are in nearly identical agreement with sc and pb results.

APPENDIXES

(Nonmandatory Information)

X1. PRECRACK CHARACTERIZATION, SURFACE CRACK IN FLEXURE METHOD

X1.1 The detectability of precracks will vary considerably between ceramic materials. Since precracks are small, of the order 0.050 to 0.200 mm (50 to 200 μm) in size, fractographic methods are needed to find and characterize them. Fractographic procedures defined in Practice C1322 and Ref (**24**) are suitable. The detectability of precracks depends upon the material, the skill of the fractographer, the type of equipment used, and the familiarity of the examiner with the material. It may be necessary to test 10 test specimens in order to obtain five precracks that are distinct. The best mode of viewing will vary from material to material. Sometimes optical microscopy is adequate, whereas, in other cases, scanning electron microscopy (SEM) is necessary. The magnifications necessary for precrack characterization are usually 100 to 500×. The superior depth of field of the scanning electron microscope is advantageous in many instances.

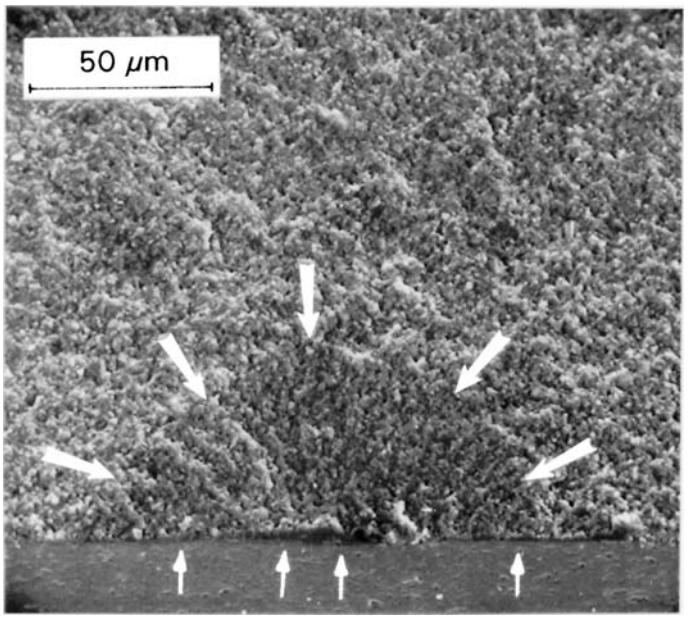
X1.2 Many ceramic materials have clear fractographic markings so that the precracks are detectable with either optical or scanning electron microscopy. Examples are shown in Figs. X1.1-X1.4. Fracture toughness measurements on the same test specimens using both optical and scanning electron microscopy precrack measurements are often in good agreement (**14, 24**). The slight differences in size measurements have only small influences on fracture toughness values, due in large part to the square root dependence of fracture toughness on precrack size.

X1.3 Many coarse-grained or incompletely-densified ceramics are not conducive to fractographic analysis. The sc method may not be suitable for these materials, since no meaningful estimate of the precrack size can be made.

X1.4 The precrack is easiest to detect if: 1) it is on a slightly different plane (angle) than the final fracture surface; 2) it fractures in a different mode (transgranular) than the final fracture (intergranular); 3) it leaves an arrest line; 4) it has been dye penetrated or thermally tinted; or 5) it has coarse or fine hackle lines which change direction at the boundary. Conditions 1, 2, or 4 will cause the precrack to have a slightly different reflectivity or contrast than the rest of the fracture surface.

X1.5 Dye penetration procedures may be beneficial for detection of the sc precrack. The optimum penetrant and impregnation procedure varies with material and some trial and error experimentation may be necessary. Experience has shown that penetration procedures work best in "white" or light-colored ceramics such as alumina and zirconia. Fluorescent penetrants may be needed for dark, opaque ceramics. Dye penetrants may not work on some materials, however. It may be difficult for dyes to penetrate the very small, very tight precracks. On the other hand, if the material has any porosity, the dyes may spread beyond the precrack. Dyes may promote slow crack growth, and therefore it is essential that they be completely dry before the fracture test. Some fluorescent dye penetrants may bleed on the fracture surface after the fracture test and extend beyond the precrack if the dye is not dry.

X1.6 Although heat treatments may be useful in highlighting or "tinting" precracks (especially in silicon carbides), this approach shall not be used in this test method since there is a



NOTE 1—No material has been removed after indenting, and portions of the Knoop indent are visible (small arrows).

FIG. X1.1 Knoop Indent Precrack in a Hot-Pressed Silicon Nitride as Photographed in a Scanning Electron Microscope

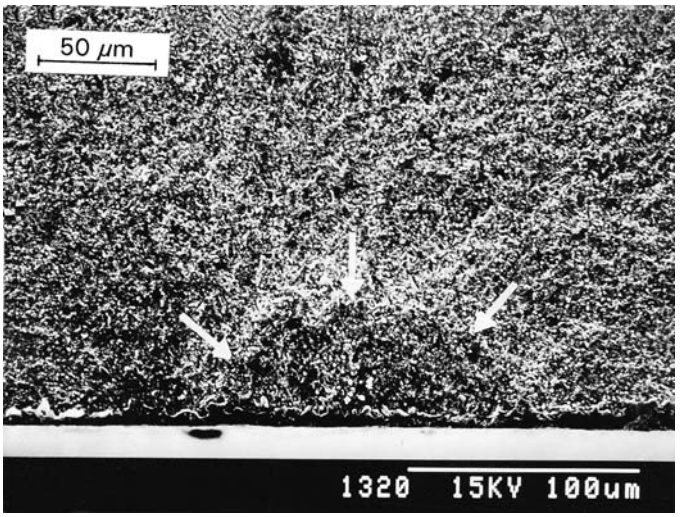
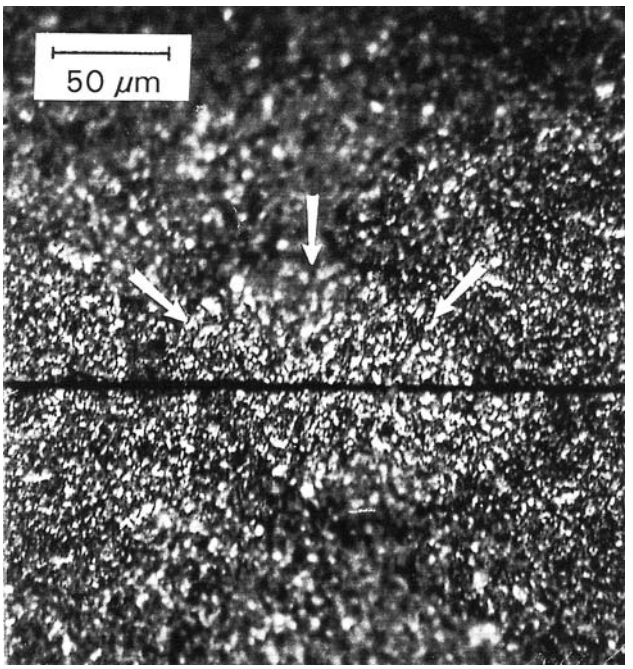


FIG. X1.2 Knoop Indent Precrack in a Hot-Pressed Silicon Nitride as Photographed in a Scanning Electron Microscope



NOTE 1—The precrack is the same as in Fig. X1.2. (Note that both halves of the test specimen are shown "back to back".)

FIG. X1.3 Optical Microscope Photograph of a Knoop Precrack in Hot-Pressed Silicon Nitride

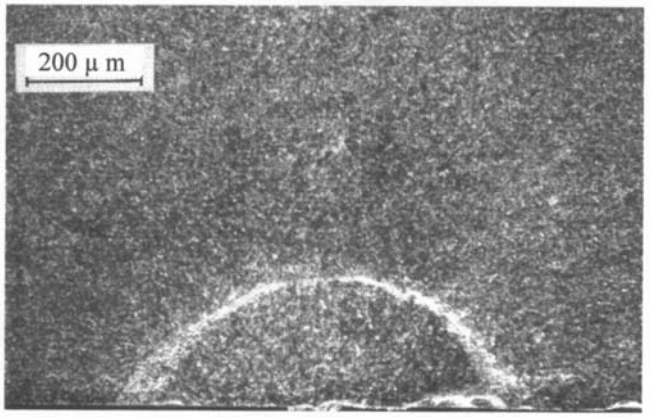


FIG. X1.4 Knoop Indent Precrack in a 99.9 % Sintered Alumina as Photographed in the Scanning Electron Microscope

risk of crack healing, crack tip blunting, or microstructural changes.

X1.7 The following paragraphs describe inspection procedures that have been effective in discerning precracks. Additional photographs and details can be found in Refs (**14, 24**).

X1.8 Both fracture surfaces should be examined. The precrack may be clearer on one surface than the other.

X1.9 Sometimes it is helpful to aim a light source at a low angle to create shadows during optical microscopy. A precrack may have a "halo" seen with either optical or electron microscopy if the crack is tilted. This is due to the different reflectivity of the ridge formed during the crack realignment to the plane of maximum stress during fracture as illustrated in Fig. X1.5. This is a purely geometrical effect. (Such markings may also be due to stable crack extension, in which case interpretation can be difficult. The guidelines of A3.5.5 are to be followed.) Reference (**35**) has additional information on precrack halos and their interpretation.

X1.10 Fine hackle lines may change direction at a boundary, and can be used to interpret the initial precrack shape as shown in Fig. X1.6. These are discernible usually only in the scanning electron microscope.

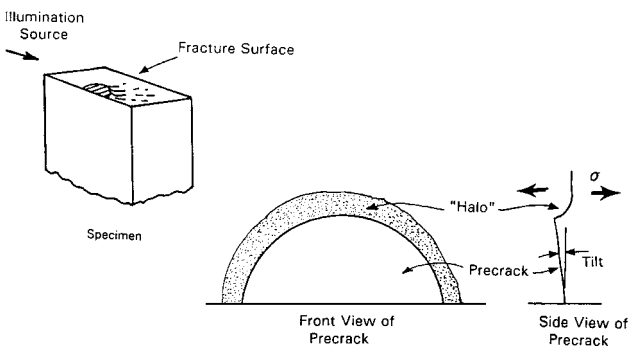


FIG. X1.5 The Slight Tilt of the Precrack can Create Shadows or Contrast Differences When Viewed in the Optical or Scanning Electron Microscope

FIG. X1.6 Fine Hackle Lines may Change Direction at the Precrack Boundary

X1.11 A combination of low- and high-power microscopy is usually very effective. This is true for both optical and electron microscopy. Lower power (50 to 100×) photographs often illustrate the precracks quite clearly, but contrast at greater magnifications is lost in the optical or electron microscope, or depth of field is lost in the optical microscope. The photograph taken at low magnification is used to find and delineate the precrack, the photograph taken at higher magnification (100 to 500×) is used for measurements of the precrack size.

X1.12 Precracks often have subtle markings which cannot be discerned on scanning electron microscope television monitors. Photography is essential with the scanning electron microscope, and will reveal precracks much better. Thermal prints should be used with caution, since experience has shown that considerable detail and clarity is lost. The thickness of the conductive coating applied to the fracture surface of the ceramic and the SEM excitation voltage may influence the contrast level between the pre-crack and the fast fracture region.

X1.13 Test specimen tilting (10 to 20°) is effective during either optical and SEM microscopy. (This is distinct from the test specimen tilt of ½ ° used during indenting). A photograph can be taken which may show the precrack quite clearly when tilted, but cannot be used for measurement due to the foreshortening of the precrack dimensions. A separate photograph taken perpendicular to the fracture surface is made for measurements, and the two photographs are compared to delineate the precrack on the latter photograph.

X1.14 Stereo photography with the scanning electron microscope is extremely effective in detecting the full topography of a precrack, and can often discern precracks quite clearly, when they are undetectable by other means. Take one photograph perpendicular to the precrack, and a second photograph at 10 to 20° off axis at the same magnification. A stereo viewer can be very helpful. Use the pair of photographs to discern the precrack, but take size measurements only from the former photograph.

X1.15 A thin gold-palladium coating, such as is used to coat nonconductive ceramics prior to electron microscope examination, can be very beneficial in optical microscopy on transparent or translucent "white" ceramics. A thin gold coating may also help with some grey ceramics such as silicon nitride. The coating can mask unwanted internal reflections and scatter. Thick gold-palladium coatings are to be avoided during coating prior to scanning electron microscopy since such coatings can obscure fine detail. A20 × 10^{-6} mm (20 nm)

coating thickness has proved effective for most ceramics. The gold-palladium coating can be applied at a shallow angle (grazing incidence) to the fracture surface. This will promote contrast which will enhance fine detail.

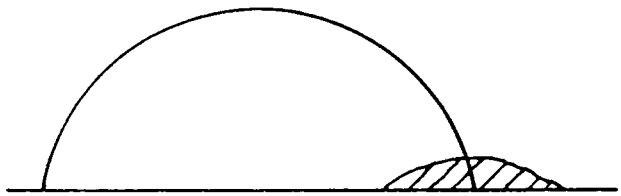
X1.16 In some instances, switching to the backscattering mode in the SEM can enhance detectability.

X1.17 In some cases, simply applying green felt tip marker ink to the fracture surface of the specimens (after fracture) helps outline the precrack. This simple step often works well on translucent or white ceramics.

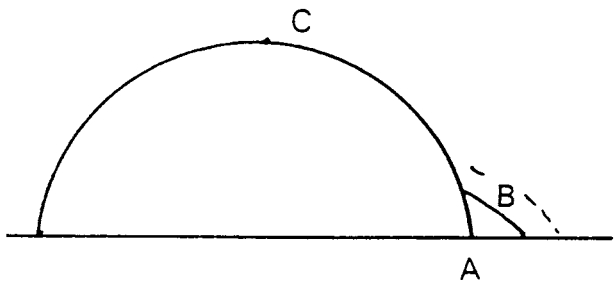
X2. COMPLICATIONS IN INTERPRETING SURFACE CRACK IN FLEXURE PRECRACKS

X2.1 Precrack interpretation may be complicated by certain features on the fracture surface. Fig. X2.1 provides guidance in such instances.

Hand Grinding or Machining Damage
This can occur if the hand grinding or machining to remove the indent is done too aggressively. Specimens with this damage can be repolished to remove the surface damage. If it is necessary to interpret such precracks, then approximate the semi-ellipse shape as if the surface damage is not present. If the maximum Y factor is at the surface, reject the datum (A3.5.3)



Corner Pop-in
During the fracture test, the precrack reaches critical fracture condition at Point A first. A small crack extends to B. Final fracture starts at Point C. The original ellipse should be used to compute fracture toughness. If the extension at points A-B is excessive, reject the datum. Hand grind the specimen more to force the Y_{max} to be at the deepest point, Y_d. (A3.5.4)



Poorly Defined Crack at the Surface
This can occur in instances where the precrack and the final fracture crack are on the same plane. (The 1/2° tilt may not have been adequate.) Alternatively, a limited depth of field in the optical microscope may hamper focusing the entire precrack. Estimate or approximate the semi-ellipse shape as best as possible, but if more than 33% of the precrack periphery is not visible, reject the datum. (A3.5.2)

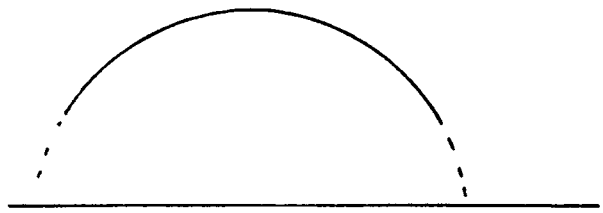
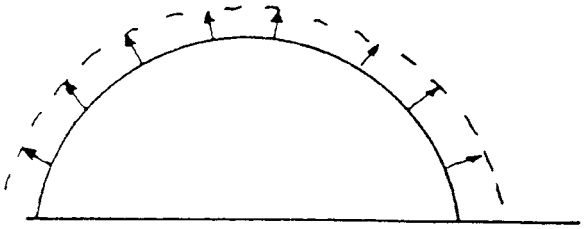


FIG. X2.1 Precrack Interpretations

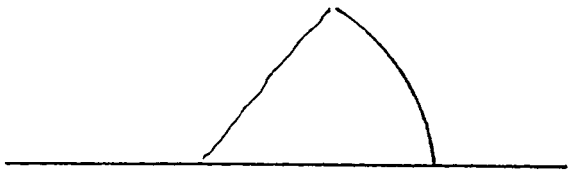
Stable Crack Extension

The crack may extend stably prior to fast fracture, either due to rising R-curve behavior, or environmentally-assisted crack growth. This can either be an interference or a useful tool to study the stable crack growth phenomena. Definitive interpretation of such stable crack extension markings on a fracture surface is extremely difficult. If stable crack extension is detected, follow the guidelines in A3.5.5 and A3.5.6.



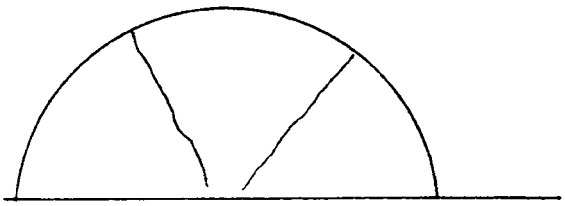
Precrack Truncation

The final crack is on a different plane and intersects only a portion of the precrack. This can occur if the precrack is not perpendicular to the maximum stress in the specimen, and fracture commences from one point on the precrack periphery, but then truncates the remainder of the precrack. In these cases, reject the datum (A3.5.2)



Precrack Segmentation

The precrack consists of three segments. The precrack is not flat and has a three-dimensional shape. It is "rippled" or "corrugated" as shown in the figure. The interference may be from lateral or Hertzian cracks associated with the original indent, or it may be due to non-uniform density in the ceramic. (This problem is common in some sintered ceramics.) If the waviness or corrugation is excessive, reject the datum. (A3.5.2)



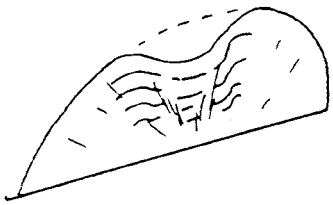
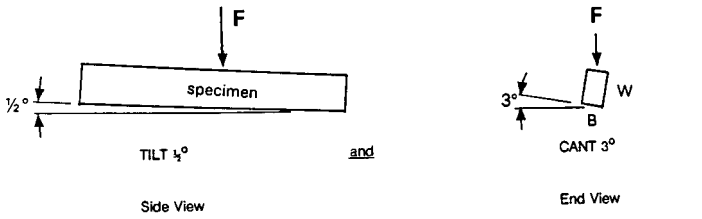


FIG. X2.1 Precrack Interpretations *(continued)*

X3. ALTERNATIVE PRECRACKING PROCEDURE, SURFACE CRACK IN FLEXURE METHOD

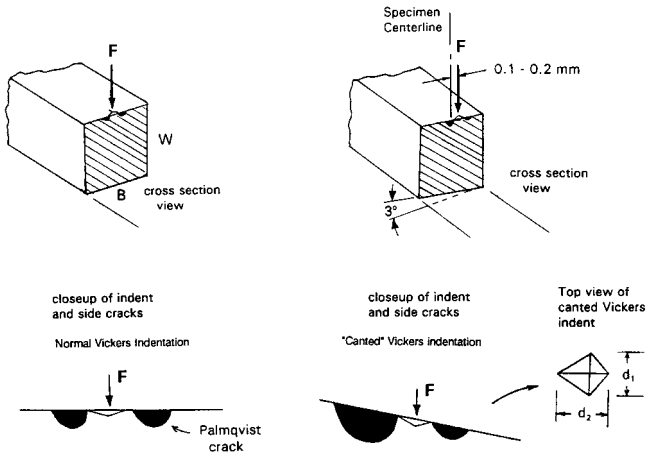
X3.1 In some very "tough" ceramics, semi-elliptical or semicircular median cracks may not form under a Knoop indent. The precracks may be very shallow and apt to be removed during the subsequent material removal steps. This can occur even if very high indent forces (for example, ~500 N) are used. In such cases, the following procedure may be used.

X3.2 Indent the polished surface of the test specimen with a Vickers indenter, taking care to orient the indent at right angles (within 2° to the test specimen long axis as shown in Fig. X3.1. Tilt and cant one end of the test specimen ½ ° and 3°, respectively, as shown in Fig. X3.1. Make the indent slightly offset from the transverse center of the test specimen surface as shown in Fig. X3.2b since the precrack that is retained after material removal is on the side of the indent. This procedure will introduce two Palmqvist cracks on the sides of



(a) Normal Vickers indent (b) Canted Vickers indent

FIG. X3.1 The Alternative Precracking Procedure for a Vickers Indenter Uses Both a Tilt and a Cant to the Test Specimen



NOTE 1—(a) Shows the Palmqvist type cracks that form on the sides of a normal Vickers indent. (b) Illustrates the cant which enlarges one side crack.

FIG. X3.2 Cross Sectional Views of SC Test Specimens Precracked by the Alternative Procedure for "Tough" Ceramics

the Vickers indent. The test specimen cant will cause one to be larger than the other. Use a full-force dwell time of 15 s or longer during the indentation cycle. Longer indentation times may be helpful for some materials such as zirconia.

X3.3 The indentation force used may have to be determined for each different class of materials through the use of a few trial test specimens. Since this alternative precracking procedure is intended for "tough" materials, greater indentation forces will be necessary (for example, 150 to 200 *N* is recommended). A single practice test specimen may be indented and broken, without the material removal steps described below in X3.4-X3.8, in order to determine whether a particular indent force is satisfactory.

X3.4 Measure the diagonals for the indent within 0.005 mm (5 μm). Calculate the average diagonal length, *d*, where $d=(d_1+d_2)/2$.

X3.5 Compute the approximate depth of the Vickers indent, *h*:

$$h = d/7 \quad \text{(X3.1)}$$

X3.6 Measure the test specimen dimension, W, in the middle of the test specimen to within 0.002 mm. A hand micrometer with a vernier graduation is suitable.

X3.7 Mark the side of the test specimen with a pencil-drawn arrow in order to indicate the surface with the precrack.

X3.8 Remove the indent and the residual stress damage zone under the indent by polishing or hand grinding to a depth of 2.5h. The procedures of A3.3.2.5 or A3.3.2.7 may be used. The precracks may be less symmetrical than those formed by the Knoop indenter and may be skewed as shown in Fig. X3.2. Knoop precracks are generally preferable since only one median precrack is formed, rather than multiple Palmqvist or median cracks associated with Vickers indents.

X4. CHAMFER CORRECTION FACTORS, SURFACE CRACK IN FLEXURE METHOD ONLY

X4.1 The fracture toughness of sc test specimens, Annex A3, should be corrected for corner chamfers if the chamfer size exceeds 0.15 mm. The chamfer size, c, shown in Fig. X4.1, may be measured with a traveling microscope, photo analysis, or a microscope with a traversing stage. All four chamfers should be measured and an average value used for the correction.

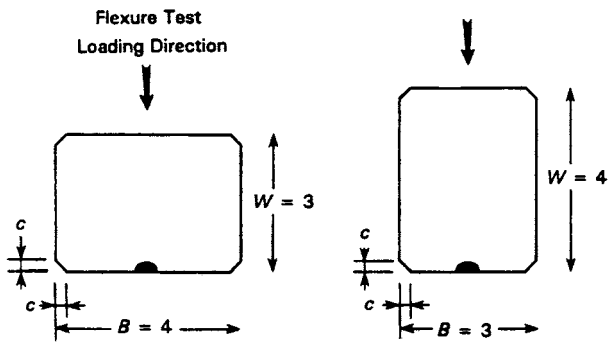


FIG. X4.1 sc Test Specimen Cross Section

X4.2 The maximum flexural stress may be calculated from simple beam theory and it is common to assume that the cross section is a simple rectangle. The chamfers alter this geometry, however, and the second moment of inertia of the test specimen cross-section about the neutral axis is altered as discussed in 36. Correction factors, F_c, for four equal chamfers are listed in Table X4.1 for test specimens with a 3 mm X 4 mm cross-section size. The factors are practically identical for the two test specimen orientations. The factors are only suitable if there are four chamfers that are of approximately equal size. Fracture toughness then may be corrected:

$$K_{Isc,cor} = F_c\, K_{Isc} \quad \text{(X4.1)}$$

X4.3 Do not correct pb or vb results for chamfers.

TABLE X4.1 Correction Factor For 3 mm X 4 mm Test Specimens

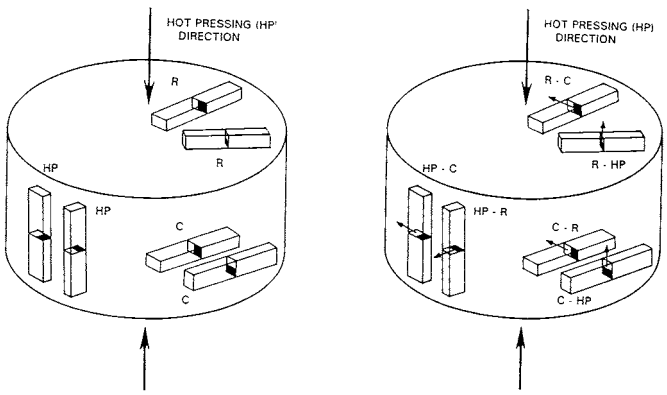
c (mm)	Correction factor, F_c B = 4, W = 3	Correction Factor, F_c B =3, W = 4
0.080	1.003	1.003
0.090	1.004	1.004
0.100	1.005	1.005
0.110	1.006	1.006
0.120	1.007	1.007
0.130	1.008	1.008
0.140	1.009	1.009
0.150	1.011	1.011
0.160	1.012	1.012
0.170	1.014	1.014
0.180	1.015	1.015
0.190	1.017	1.017
0.200	1.019	1.019
0.210	1.020	1.021
0.220	1.022	1.023
0.230	1.024	1.025
0.240	1.027	1.027
0.250	1.029	1.030

X5. CRACK ORIENTATION

X5.1 The crack orientation is a description of the plane and direction of a fracture in relation to a characteristic direction of the product. At the minimum, record and report the plane of fracture. Ideally report both the plane of fracture and the direction of crack propagation. The characteristic direction may be associated with the product geometry or with the microstructural texture of the product.

X5.2 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 hotpressing, hot isostatic pressing).

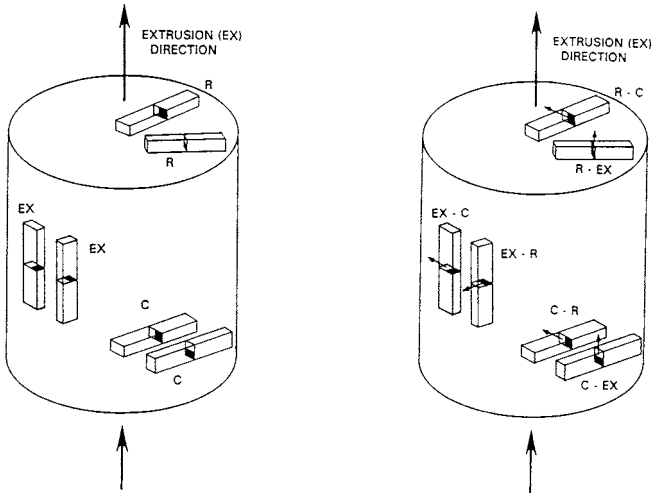
X5.3 The crack plane is defined by letter(s) representing the direction normal to the crack plane as shown in Fig. X5.1a and Fig. X5.2a. For a rectangular product, R and C may be replaced by rectilinear axes x and y, corresponding to two sides of the plate. Isopressed products, amorphous ceramics, glasses 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



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

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

FIG. X5.1 Crack Plane Orientation Code for Hot-Pressed Products



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

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

FIG. X5.2 Crack Plane Orientation Code for Extruded Products

direction of the product. Use the same designation scheme as shown in Fig. X5.1 and Fig. X5.2, but with the letters "AXL" to denote the axial axis of the product. If there is no primary product direction, reference axes may be arbitrarily assigned but must be clearly identified.

HP = hot-pressing direction (See Fig. X5.1)
EX = extrusion direction (See Fig. X5.2)
AXL = axial, or longitudinal axis (if HP or EX are not applicable)
R = radial direction (See Fig. X5.1, Fig. X5.2)
C = circumferential direction (See Fig. X5.1, Fig. X5.2)
R/C = mixed radial and circumferential directions

X5.4 The crack direction may also be designated and is defined by the letter(s) representing the direction parallel to the characteristic direction (axis) of the product as illustrated in Fig. X5.1b and Fig. X5.2b. 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.

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