
**Fine ceramics (advanced ceramics,
advanced technical ceramics) —
Determination of fracture toughness of
monolithic ceramics at room temperature
by the surface crack in flexure (SCF)
method**

*Céramiques techniques — Détermination de la ténacité à la rupture des
céramiques monolithiques à température ambiante par fissuration
superficielle en flexion*



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 18756 was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

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Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of fracture toughness of monolithic ceramics at room temperature by the surface crack in flexure (SCF) method

1 Scope

This International Standard describes a test method that covers the determination of fracture toughness of monolithic ceramic materials at room temperature by the surface crack in flexure (SCF) method.

This International Standard is intended for use with monolithic ceramics and whisker- or particulate-reinforced ceramics that are regarded as macroscopically homogeneous. It does not include continuous-fibre reinforced ceramic composites.

The test method is applicable to materials with either flat or rising crack growth resistance curves. This method is similar to ISO 15732 except that precracks are smaller and are made by a different procedure. The methods should produce similar or identical results for materials with a flat R-curve.

NOTE This test method is usually applicable to ceramic materials with a fracture toughness less than $\approx 10 \text{ MPa m}^{1/2}$. It may be difficult to form precracks with a Knoop indenter for materials with greater fracture toughness or those materials which are soft (low hardness) such as some zirconias, or for porous ceramics.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3611:1978, *Micrometer callipers for external measurement*

ISO 7500-1:—¹⁾ *Metallic materials — Verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Verification and calibration of the force-measuring system*

ISO 14704:2000, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for flexural strength of monolithic ceramics at room temperature*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

1) To be published. (Revision of ISO 7500-1:1999)

**3.1
stress intensity factor**

K_I
magnitude of the elastic stress field singularity at the tip of a crack subjected to opening mode displacement

NOTE It is a function of applied force and test specimen size, geometry and crack length, and has dimensions of force times length to the power of three over two.

**3.2
fracture toughness**

generic term for measures of resistance of extension of a crack

**3.3
fracture toughness value**

K_{Isc}
fracture toughness value measured by the SCF method

NOTE This is the measured stress intensity factor corresponding to the crack extension resistance of a semi-elliptical small crack formed underneath a Knoop indentation. The measurement is performed to the operational procedure herein and satisfies all the validity requirements.

**3.4
precrack**

crack introduced into the test specimen artificially prior to testing the specimen to fracture

**3.5
crack front line**

line to indicate the position of the tip of the crack

**3.6
critical stress intensity factor**

K_{Ic}
critical value of K_I at which fast fracture occurs

**3.7
critical crack**

crack at fracture at maximum load and whose stress intensity factor just reaches the critical stress intensity factor

**3.8
critical crack size**

size of the critical crack at fracture

NOTE The critical crack will be larger than the precrack if stable crack extension occurs due to environmentally-assisted slow crack growth or rising R-curve behaviour.

**3.9
four-point 1/4-point flexure**

specific configuration of four-point flexural strength testing where the inner bearings are situated one quarter of the support span away from the two outer bearings

**3.10
four-point 1/3-point flexure**

specific configuration of four-point flexural strength testing where the inner bearings are situated one third of the support span away from the two outer bearings

**3.11
flexural strength**

maximum nominal stress at fracture of a specified elastic beam loaded in bending

4 Symbols

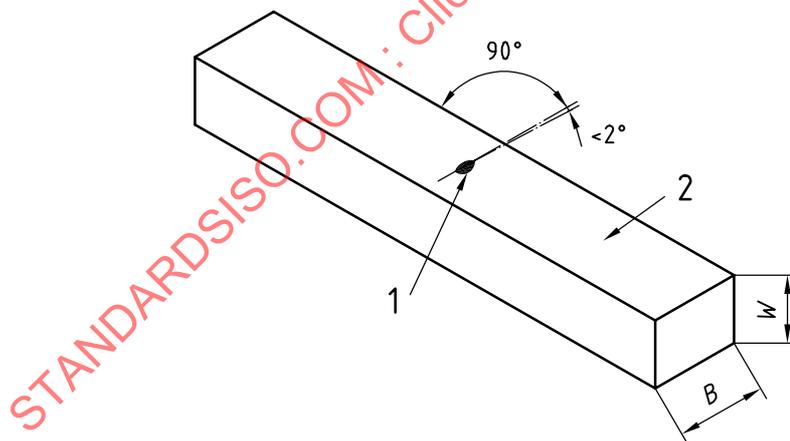
a	Crack depth
A	Flexure fixture moment arm
B	Specimen width, the cross section dimension perpendicular to the direction of loading in bending
c	Crack half width
C	Chamfer size
d	Length of Knoop indentation long diagonal
h	Depth of Knoop indentation
F	Knoop indentation load
F_c	Chamfer correction factor
$H_1(a/c, a/W)$	A polynomial in the stress intensity factor coefficient, for the point on the crack periphery where it intersects the specimen surface
$H_2(a/c, a/W)$	A polynomial in the stress intensity factor coefficient, for the deepest part of the surface crack
K_I	Stress intensity factor, Mode I
K_{Ic}	Critical stress intensity factor, Mode I
K_{Isc}	Fracture toughness value, surface crack in flexure method
L	Flexure fixture support span
L_T	Specimen length
$M(a/c, a/W)$	A polynomial in the stress intensity factor coefficient
P	Load at fracture
$Q(a/c)$	A polynomial function of the surface crack ellipticity
$S(a/c, a/W)$	Factor in the stress intensity factor coefficient
W	Specimen depth, the cross section dimension parallel to the direction of loading in bending
Y	Stress intensity factor coefficient
Y_d	Stress intensity factor coefficient at the deepest part of the surface crack
Y_{max}	The maximum stress intensity factor coefficient along the boundary of the surface crack
Y_s	Stress intensity factor coefficient at the intersection of the surface crack with the specimen surface

5 Principle

This International Standard is for material development, material comparison, quality assurance, characterization, reliability, and design data generation. The method determines the fracture toughness value, K_{Isc} by fracturing a common flexure specimen which has a small surface precrack (see Figure 1). The specimen is indented with a Knoop indenter in order to make a small, semi-elliptical surface crack. The specimen is polished or ground carefully until the indentation and associated residual stress field are removed. The specimen is fractured in four-point flexure. The fracture toughness, K_{Isc} , is calculated from the fracture load and the measured critical crack size. Fractography is required to measure the precrack size and to determine whether the crack has grown in size. Fracture toughness as a function of crack size may be evaluated by varying the Knoop indentation load that is used to make the precrack. Background information concerning this test method may be found in References [1] and [2]. An international interlaboratory comparison study (round robin) project on this method is described in References [3], [4] and [5].

If the ceramic is too soft (low hardness) or has too great a fracture toughness, it may be difficult to create a precrack by the SCF method. In addition, for some materials (particularly those with coarse grain or heterogeneous microstructures), it may be difficult to detect the crack on the fracture surface. If the user is not sure of the applicability of this method, then a single trial specimen may be tested with an abbreviated procedure. Indent the specimen and fracture it without removal of the indentation and residual stresses. Inspect the fracture surface to confirm that the specimen fractured from the precrack (and not from a material flaw) and that the precrack can be detected on the fracture surface.

Precracking is by Knoop indentation only in this International Standard. Residual stresses underneath the indentation are removed in this test method. There is some limited experience with SCF precracking by Vickers indentation [3, 4, 5, 6, 7]



Key

- 1 indentation and precrack
- 2 polished or lapped surface

Figure 1 — Indentation and precrack in a flexural specimen

6 Apparatus

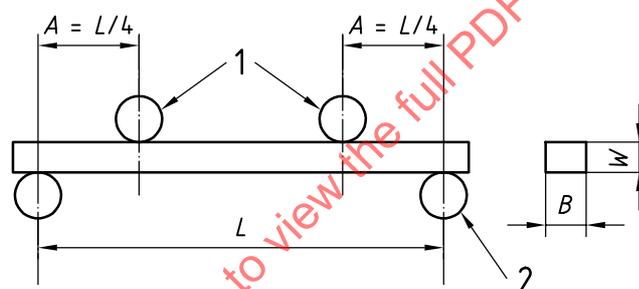
6.1 Testing machine, capable of applying a uniform cross-head speed. The testing machine shall be in accordance with ISO 7500-1:— Class 1 with an accuracy of 1 % of indicated load at fracture.

6.2 Flexural fixtures, four-point as shown in Figure 2. Flexural fixtures shall meet the requirements of ISO 14704.

The fixtures shall either be semi-articulating or fully-articulating depending upon the condition of the specimens. If the specimens meet the parallelism requirements of 7.1, then semi-articulating fixtures may be used. Semi-articulating fixtures are usually completely satisfactory for machined specimens. If the specimens do not meet the parallelism requirements of 7.1 (due to hand grinding unevenness, problems with machining, or other causes), then fully-articulating fixtures shall be used. Fully-articulating fixtures also may be used with machined specimens. Specimens shall be loaded and supported by bearings. The bearings shall be free to roll in order to eliminate friction. For four-point flexure, the two inner bearings shall be free to roll *inwards*, and the two outer bearings shall be free to roll *outwards*.

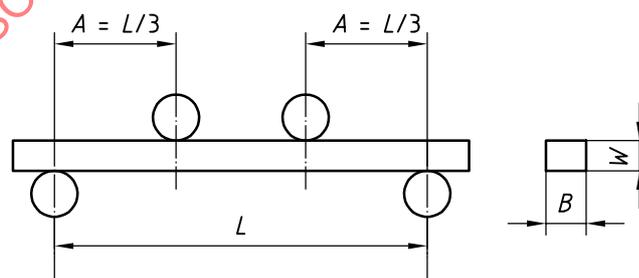
Four-point 1/4-point fixtures with 20 mm support span and 10 mm inner span are also permitted. Such fixtures shall meet the requirements of ISO 14704.

Dimensions in millimetres



$$L = 40,0 \pm 0,10 \text{ or } L = 20,0 \pm 0,10$$

a) Four-point 1/4-point flexure



$$L = 30,0 \pm 0,10$$

b) Four-point 1/3-point flexure

Key

- 1 loading bearings
- 2 support bearings

Figure 2 — Four-point flexure

6.3 Micrometer, such as described in ISO 3611 but with a resolution of 0,002 mm shall be used to measure the specimen dimensions. The micrometer shall have flat anvil faces such as shown in ISO 3611. The micrometer shall not have a ball tip or sharp tip since these might damage the specimen. Alternative dimension measuring instruments may be used provided that they have a resolution of 0,002 mm or finer.

6.4 Hardness testing machine, conventional type, to create the Knoop indentation. The machine shall be able to apply loads of 20 N to 50 N or greater. If a hardness machine with this load range is not available, then a strength testing machine (6.1) may be used although some loss of accuracy or control of indentation and crack size may result.

6.5 Microscopes, optical and/or scanning electron, shall be used to detect the precrack (or critical crack) and measure its size on the specimen fracture surface after the test. Magnifications of 100× to 500× are usually required. The microscope shall be capable of making photographic or digital records of the cracks.

6.6 Dye penetrants, to highlight the crack. Dye penetrants that do not promote slow crack growth nor bleed (spread on the fracture surface after fracture) are preferred.

6.7 Temperature measuring device, a thermometer or other device to measure ambient temperature during the fracture of the specimen.

6.8 Humidity measuring device, such as a hygrometer, sling psychrometer, or other device to measure ambient humidity during the fracture of the specimen.

7 Test specimens

7.1 Specimen size, preparation and edge chamfering

7.1.1 Rectangular beam specimens with dimensions as shown in Figure 3 shall be used. The cross-sectional tolerances are $\pm 0,2$ mm. The parallelism tolerance on opposite longitudinal faces is 0,015 mm.

7.1.2 Specimens shall be prepared in accordance with ISO 14704 with the exceptions noted below. The indentation may be placed in either a 3 mm or 4 mm wide face. A diamond-grit wheel 320 or finer shall be used to remove the last 0,04 mm on the surface that is used for the indentation. This surface shall be polished, lapped or fine ground to provide a flat, smooth surface for the surface crack.

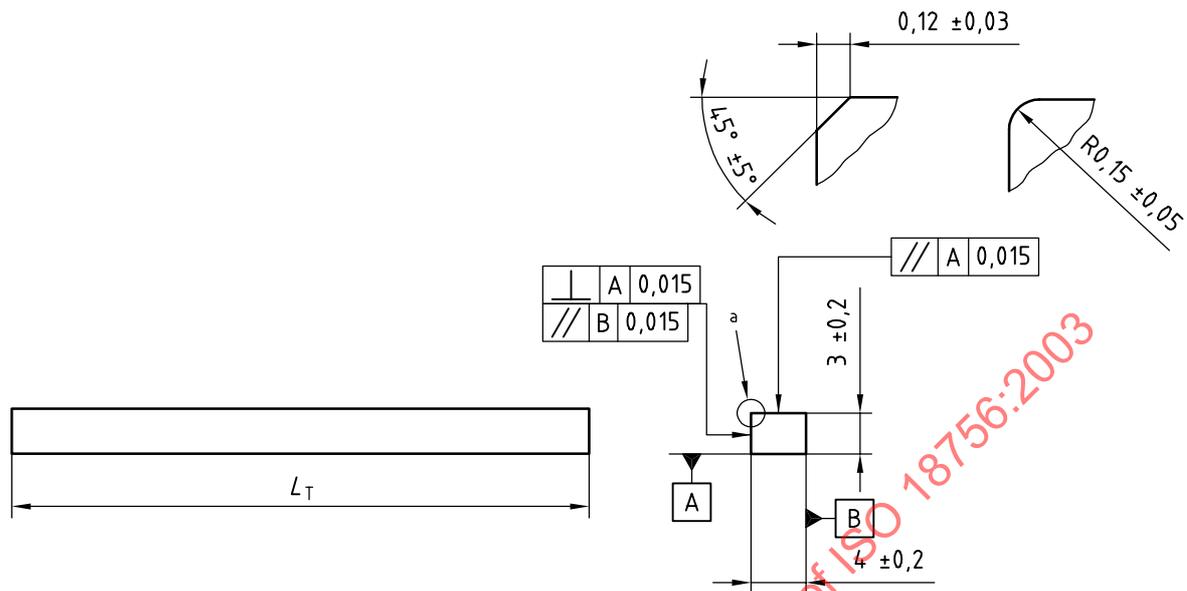
NOTE The surface does not require a polished, high-quality finish such as required for a hardness measurement. The surface need only be flat so that the Knoop indentation is not affected by machining striations or marks, or specimen unevenness.

7.1.3 Chamfers or edge rounds are optional. If premature fracture occurs from edge damage, then the edges shall be chamfered or rounded as specified in ISO 14704. The chamfer size should be 0,15 mm or less. See Figure 3.

7.2 Number of specimens

The number of specimens shall be not less than five. It is recommended that at least ten specimens be prepared. This will provide specimens for practice tests to determine the best indentation loads and provide specimens to make up for unsuccessful or invalid tests. More specimens are needed if environment, testing rate, or precrack sizes are to be varied.

Dimensions in millimetres



$L_T \geq 25$ for 20 mm test fixtures or
 $L_T \geq 35$ for 30 mm test fixtures or
 $L_T \geq 45$ for 40 mm test fixtures

^a Edge chamfers or rounding.

Figure 3 — Test specimen dimensions

8 Procedure

8.1 Introduction of the precrack by Knoop indentation

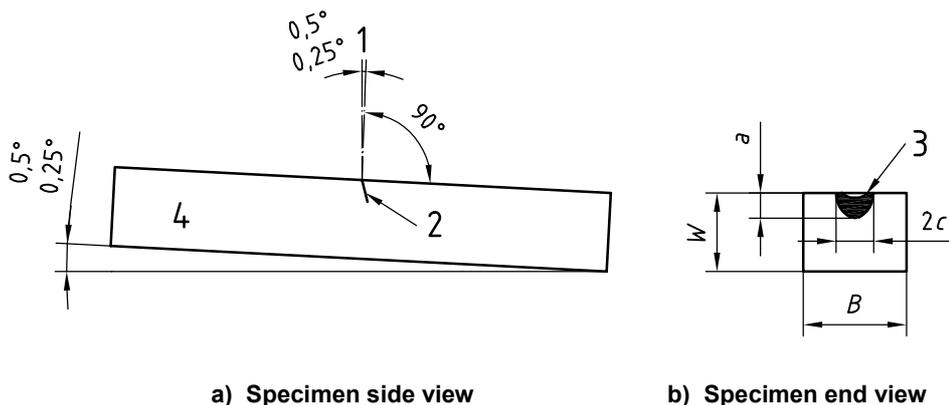
8.1.1 Use a Knoop indenter to indent the middle of the polished, lapped or fine ground surface of the specimen. The indentation shall be perpendicular to the specimen long axis to within 2° as shown in Figure 1. One end of the specimen shall be tilted approximately $0,25^\circ$ to $0,5^\circ$ as shown in Figure 4. A full load dwell time of 15 s or more during the indentation cycle shall be used. The indentation may be placed in either a 3 mm or 4 mm wide face as shown in Figure 5. It is recommended that the indentation be placed near to the exact centre of the surface, both along the width dimension B and along the specimen length, in order to make it easy to confirm that fracture occurs from the precrack.

NOTE 1 The $0,25^\circ$ to $0,5^\circ$ tilt makes the precrack easier to detect on the fracture surface. The specimen tilt causes precrack tilts from 0° to 5° .

NOTE 2 In some instances such as with zirconia, indentation times longer than 15 s may be helpful.

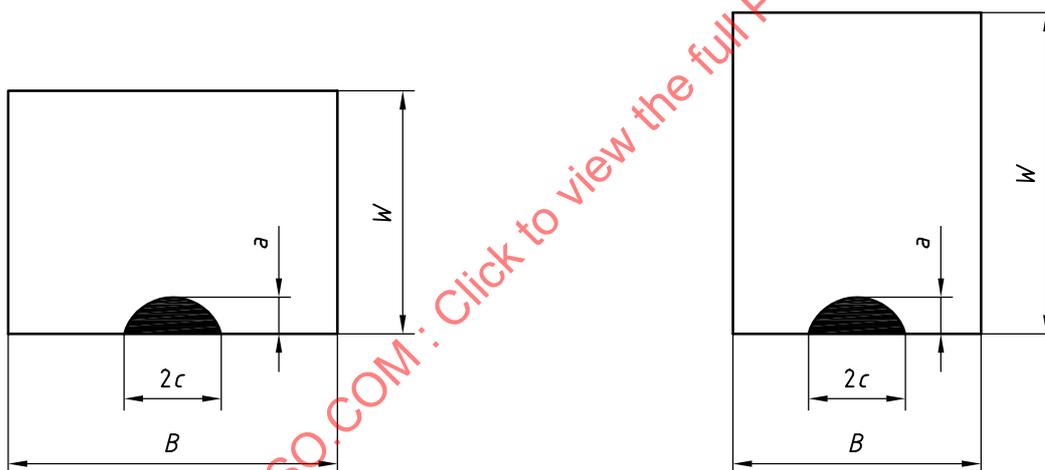
NOTE 3 A trial specimen may be tested to help determine the best indentation load. Indent then fracture the specimen in the flexure fixtures without removal of the indentation and residual stress damage zone. Examine the fracture surface to confirm that the specimen has fractured from the precrack, that the precrack is discernible, and within the prescribed size limits.

NOTE 4 The Knoop indentation procedure to create a surface crack will not be successful on very soft or porous ceramics since a precrack will not form under the indentation. The process may not work on very tough ceramics which are resistant to the formation of cracks, or where the cracks are very small and likely to be removed during the subsequent polishing step to remove the residual stress and damage zone.



- Key**
- 1 Knoop indenter
 - 2 precrack
 - 3 indent with precrack
 - 4 platform tilts specimen

Figure 4 — Indentation of the precrack by Knoop indentation



NOTE 1 The indentation may be placed on either the wide 4 mm face or the narrow 3 mm face.

NOTE 2 The crack size has been exaggerated for illustrative purposes and is usually much smaller.

Figure 5 — Specimen cross section

8.1.2 The optimum indentation load used may have to be determined for each different class of material by the use of a few trial specimens. The load shall be sufficient to create a crack that is larger than the naturally-occurring flaws in the material, but not too large relative to the specimen cross section size ($2c < 0,5B$ and $a < 0,5W$) nor so large that the indentation is badly spalled or shattered. Indentation loads of approximately 20 N are suitable for very brittle ceramics, 25 N to 50 N for medium tough ceramics, and 49 to 98 N for very tough ceramics or ceramics with some porosity. Indentation loads of 98 N to 147 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.

8.1.3 Measure the length of the long diagonal, d , of the Knoop impression to within 0,005 mm (5 μ m).

NOTE A conventional microhardness machine (6.4) may be used for this measurement. The measurement does not require the precision needed for hardness measurements. If the Knoop hardness is reported, greater care is recommended in making the diagonal size measurement and in the preparation of the initial specimen surface.

8.1.4 Compute the depth, h , of the Knoop impression as follows:

$$h = d / 30 \quad (1)$$

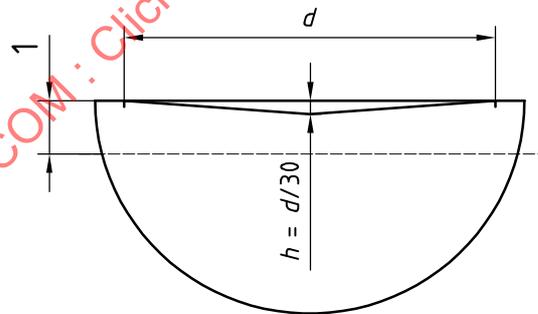
8.1.5 Measure the specimen depth, W , in the middle of the specimen at the indent location to within 0,002 mm. A hand micrometer (6.3) with vernier graduations marked in 0,002 mm increments is suitable.

8.1.6 Mark the side of the specimen with a pencil or other marker with an arrow to indicate which surface has the precrack.

8.1.7 Remove the indentation and the residual stress damage zone.

8.1.7.1 Remove from the indented surface an amount of material that is approximately equal to $4,5h$ to $5,0h$ as shown in Figure 6. The material removal process shall not induce residual stresses or excessive machining damage in the specimen surface. Be careful to remove material from the correct face. Mark with pencil or permanent marker the faces that will not be ground or polished. Material may be removed by any one of the three procedures described in 8.1.7.2, 8.1.7.3 and 8.1.7.4.

NOTE The removal of $4,5h$ to $5,0h$ eliminates the residual stress damage zone under the impression, and usually will leave a precrack shape that has the greatest 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 the maximum Y , stress intensity factor coefficient, 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} will shift 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 it is preferable to remove smaller amounts, provided that no less than $3h$ is removed. If this step is not adequate to ensure fracture from the precrack, then a greater indentation load may be needed.



Key

1 material to be removed after indentation

NOTE The precrack extends below the Knoop hardness indentation, which has a depth h .

Figure 6 — The indentation and the residual stress damage zone

8.1.7.2 Material may be removed by hand grinding, hand lapping or hand polishing with abrasive papers under wet or dry conditions. Hand polishing the specimen with 180 to 220 grit silicon carbide paper can remove the required amount in 5 min to 10 min per specimen for many ceramics. Check the specimen height frequently during this process. Remove the last 0,005 mm with a finer grit (220 to 280 grit) paper with less pressure, so as to minimize polishing damage. Monitor the specimen depth, W , frequently during the material removal step, with special emphasis on monitoring the evenness of the material removal.

Hand grinding, hand lapping or hand polishing may not be effective with very hard ceramics. For very hard ceramics, material may be removed by machine polishing or lapping (8.1.7.3), or by machine surface grinding (8.1.7.4).

Regularly change the orientation of the surface being hand polished or ground during material removal in order to minimize unevenness. Unevenness may cause misalignments during subsequent flexure testing or cause errors in the cross section size measurements.

NOTE 1 Dry hand grinding may be faster than wet grinding. Diamond impregnated polishing discs (30 µm) are also effective in removing material by hand grinding.

NOTE 2 Hand lapping or grinding may make the precracked surface uneven or not parallel to the opposite specimen surface. A slight rounding of the specimen edges is usually inconsequential.

NOTE 3 Finer grit (320 to 500 grit) papers are recommended for glasses for both rough removal and fine finishing steps

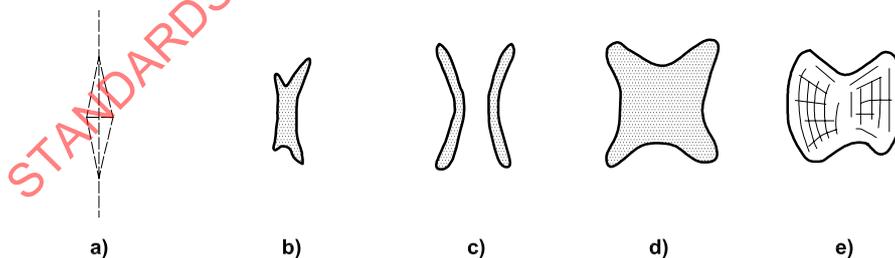
CAUTION — Fine ceramic powders or fragments may be created if the lapping or hand sanding is done dry. Masks should be used or the removal done wet if there is an inhalation hazard, especially if the ceramic contains silica or fine whiskers.

8.1.7.3 Material may be removed by machine polishing or lapping with diamond slurry or paste containing about 0,3 µm particles. This requires about 10 min to 15 min per specimen for many ceramics. A trial specimen may be polished to obtain an appropriate removal rate by adjusting applied masses, rotating speed of the disc and polishing times in order to obtain the correct amount of material removal.

8.1.7.4 Material may be removed by surface grinding with diamond wheels on a grinding machine for very hard materials. Take care to ensure that the correct amount of material has been removed from each specimen. Avoid aggressive grinding conditions that may introduce residual stresses. If machine surface grinding is used, fine wheel grits (320 to 600 grit) and small removal rates are recommended. Grinding may be carried out under wet conditions.

8.1.7.5 After the prescribed amount of material has been removed, examine the ground-indentated surface for evidence of remnant lateral cracks. Figure 7 provides guidance. A low power reflected light metallurgical optical microscope (6.5) with magnifications from 100× to 500× may be used to examine the ground-indentated tensile surface. If there is evidence of remnants of lateral cracks, then additional material should be removed (6h to 10h) to ensure that the lateral cracks remnants are removed.

NOTE Deeper than normal lateral cracks may occur in materials with very low fracture toughness (< 3,0 MPa m^{1/2}) or if larger indentation loads (≥ 98 N) are used.



a) shows the ground surface after material removal. The Knoop indentation (dashed lines) has been removed and the median crack is very tight and not visible. There are no traces of lateral cracks.

b) to e) show examples of remnants of lateral cracks which should be removed in accordance with 8.1.7.4 and 8.1.7.5.

Figure 7 — A ground surface and remnants of lateral cracks existing after removal of the damage zone in some brittle materials

8.1.7.6 Annealing or heat treating to remove the residual stresses under the indentation is not permitted by this International Standard due to the risk of crack tip blunting or crack healing.

8.1.8 Dry the specimen prior to testing if the material removal is done wet.

NOTE There is no consensus on the best conditions for drying specimens. Heating in an air or vacuum oven at 100 °C to 150 °C for times up to 1 h and then storage in a desiccator prior to testing may be sufficient.

8.1.9 If necessary, a dye penetrant (6.6) may be applied to aid crack detection. If a dye penetrant is used, the specimen should be dried thoroughly prior to fracture.

8.1.10 Measure and record the specimen dimensions, B and W , in the vicinity of the precrack to within 0,002 mm.

8.2 Specimen fracture

8.2.1 Ensure that the specimen is dry.

8.2.2 Test the precracked specimens to fracture in four-point flexure in laboratory ambient conditions. If the material is susceptible to slow crack growth, it is recommended that the testing conditions of 8.4 be applied.

NOTE Many oxides, glasses and ceramics having glassy boundary phases may be susceptible to slow crack growth. Measured fracture toughness may be sensitive to displacement rate and moisture in the atmosphere. See Annex A for additional background information.

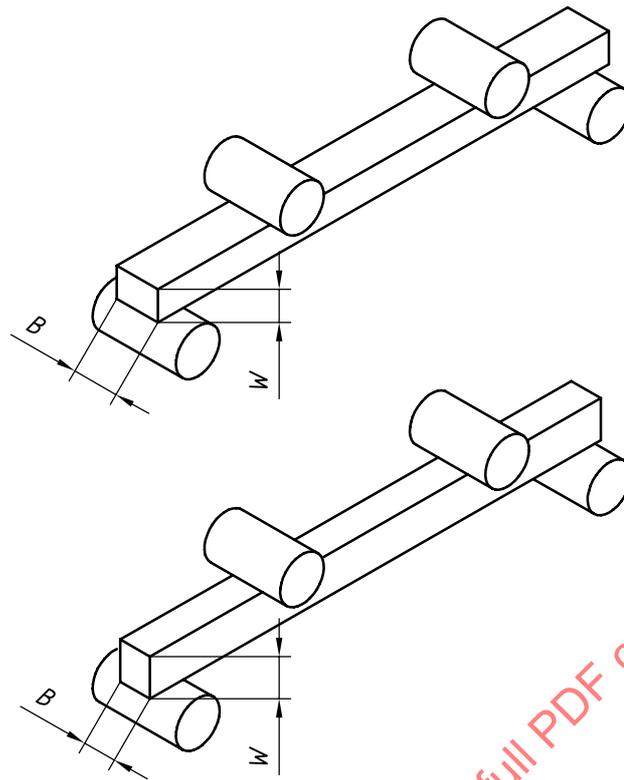
8.2.3 Insert the specimen into the flexure fixture as shown in Figure 8, with the surface crack on the tension face, approximately in the middle (within 1 mm) of the two inner loading rollers. The specimen may be preloaded to no more than 25 % of the expected fracture load. Place cotton, crumpled tissue or other appropriate material under the specimen to prevent the pieces from impacting the fixture upon fracture and to prevent the fracture surface from being damaged by impact after the specimen breaks. Place a simple shield around the fixture to ensure operator safety and to preserve the primary fracture pieces for subsequent fracture analysis. If the specimen precracked face is not parallel to the opposite face to within 0,015 mm, then fully-articulating fixtures shall be used.

8.2.4 Use a standard displacement rate of 0,5 mm/min for specimens tested with 30 mm or 40 mm support span fixtures. Use a displacement rate of 0,10 mm/min to 0,13 mm/min for specimens tested with 20 mm support span fixtures.

8.2.5 Apply a compressive load to the fixture until the specimen fractures. Measure the fracture load to an accuracy of ± 1 %. The time to fracture should not exceed 20 s in order to minimize environmental effects. If the time to fracture is greater than 20 s, use a faster displacement rate than those given in 8.2.4.

8.2.6 Measure the ambient temperature during the test series.

8.2.7 Measure the ambient relative humidity during the test series if tests are performed in laboratory ambient conditions.



The flexure specimen may be tested with either the wide or narrow face on the loading bearings. The precrack shall be on the tension surface which is the bottom surface in this figure.

Figure 8 — The flexural specimens on the loading bearings

8.3 Crack size measurement

8.3.1 Examine the fracture surface of the specimens and measure the critical crack dimensions a and $2c$ as shown in Figure 4 or 5. Fractographic techniques and fractographic skill are needed for this step. 8.3.2 to 8.3.10 give more detail on the procedures that may be used. Annex B provides guidance on crack detection and characterization. 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, $2c$, to within 0,010 mm (10 μm) or less if possible.

NOTE The achievable precision of the crack size measurement depends upon the material and its microstructure, the clarity of the crack and the mode of viewing. For some materials, it is possible to measure the crack size with greater precision than suggested in 8.3.1, but in other materials the achievable precision may be less than suggested in 8.3.1. In many instances, the computed fracture toughness is not very sensitive to the precision of the crack size measurement as discussed in References [3] and [5]. 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 in 8.3.1.

8.3.2 The optimum procedure will vary from material to material. Either an optical microscope or a scanning electron microscope, or both may be used. Low magnifications (50 \times to 100 \times) may be used to locate the crack, and higher magnifications (100 \times to 500 \times) to directly measure or photograph the crack for measurement.

8.3.3 If an optical microscope is used, then variation of the lighting source direction can be used to highlight the crack. Stereo binocular optical microscopes are preferred to metallographic microscopes. Crack sizes may be measured from photos taken of the fracture surface, by direct measurement while viewing the specimen if the microscope has a precision transversing stage for the specimen, or by an eyepiece filar measurement device. If photos are taken, the fracture surface plane should be normal to the camera axis and a stage micrometer should be used to confirm the magnifications.

8.3.4 If a scanning electron microscope (SEM) is used, then an SEM magnification calibration standard shall be used to confirm the magnification.

NOTE Additional details on techniques to find and characterize the cracks for both optical and SEM microscopy are given in Annex B.

8.3.5 The crack shape may be approximated by a semi-ellipse. This approximation is most accurate for instances where the greatest stress intensity factor coefficient is at the deepest part of the crack ($Y_{\max} = Y_d$; see 9.1). If the maximum stress intensity factor coefficient is at the surface ($Y_{\max} = Y_s$), then re-examine the crack shape to confirm that the crack is semi-elliptical. If it is not, then reject the datum.

8.3.6 If the crack form is severely distorted in the third dimension (i.e. is not flat), or the crack front line is incomplete over more than 33 % of its periphery, reject the datum. See Figures B.7 c), B.7 e) and B.7 f).

8.3.7 If hand grinding or machining damage [see Figure B.7 a)] interfere with the determination of the crack shape and $Y_s > Y_d$, then reject the datum.

8.3.8 If the precrack shows evidence of excessive extension (corner pop-in) at the intersection of the surface [see Figure B.7 b)], then reject the datum.

8.3.9 If the precrack shows evidence of stable crack extension prior to fracture, then measure both the initial precrack size and the critical crack size. [See Figures B.4 and B.7 d)].

8.3.10 If the crack width is such that $2c > 0,5B$ or the depth is such that $a > 0,5W$, then reject the datum. A smaller indentation load may be used.

8.4 Environmental effects

8.4.1 If susceptibility to environmental degradation, such as slow crack growth, is a concern, then tests should be performed in accordance with either 8.4.2, 8.4.3 or 8.4.4.

8.4.2 Perform tests at two different displacement rates. The two test rates should differ by at least two or three orders of magnitude. One rate should be very slow, so that the crack has a chance to react to the environment. Susceptibility may be evaluated by comparing the mean fracture toughness values at the two rates. Environmental susceptibility may also be determined by examining the fracture surfaces for evidence of crack extension such as halos at the slower testing rate [Figures B.4 and B.7 d)]. If the material is susceptible to environmental effects, then determination of the critical crack size is required. Annex A provides some examples of the use of the critical crack sizes and also variations of displacement rate. If the precrack size or incorrect critical crack size is used for the calculation, the fracture toughness values may strongly depend on the displacement rate.

8.4.3 Perform tests in an inert environment such as dry nitrogen gas. Select an atmosphere which is considered not to adversely affect the crack growth of the specimen during the flexure test. Recommended atmospheres include dry air, nitrogen or argon with a purity of 99,9 % or better at atmospheric pressure, or a vacuum of less than 0,13 Pa. Alternatively, specimens may be coated with a paraffin wax. Use a displacement rate of 0,5 mm/min.

NOTE 1 For inert atmosphere testing, a simple chamber around the test fixtures or even a sealed plastic bag may be adequate, provided that the laboratory ambient air can be flushed for several minutes between tests.

NOTE 2 If paraffin wax is used, then avoid contamination of the fracture surface and the precrack after fracture.

8.4.4 Perform one set of experiments in normal laboratory ambient conditions (8.2.2, 8.2.3, and 8.2.4) and one set of experiments in an inert atmosphere (8.4.3). Environmental susceptibility may be determined by comparing the mean fracture toughness for the two data sets. Environmental susceptibility may also be determined by comparing the fracture surfaces and determining whether the laboratory ambient tested specimens show evidence of crack extension. If environmental sensitivity is detected, use the results from the inert experiments to compute the fracture toughness. The normal laboratory ambient results may also be used if the critical crack size is detected.

8.4.5 Fracture toughness, K_{Isc} , should be the fracture toughness for which environmental effects are eliminated or minimized. If slow crack growth susceptibility is detected, then report the fracture toughness determined in inert atmosphere, or the fracture toughness at the fastest loading rates and whenever possible, the fracture toughness based upon the critical crack size.

8.5 Optional: Estimate of R-curve behaviour

An estimate of the effect of crack size upon fracture toughness (for an assessment of possible R-curve effects) may be obtained by any one of several methods described in Annex C.

8.6 Optional: Reference materials

Reference materials may be used to verify the procedures in this test method.

9 Calculation

9.1 Calculate the stress intensity shape factor coefficients for both the deepest point of the crack periphery, Y_d , and for the surface, Y_s . Use the critical crack size dimensions.

NOTE The stress intensity factors are from Reference [8] and are only strictly valid for $a/c \leq 1$, $a/W < 1,0$ and $2c/B < 0,5$. They may be used for a/c ratios slightly greater than 1 with a slight loss of accuracy according to Reference [9]. For most practical cases, the precracks are much smaller than the cross section dimensions B or W .

For the deepest point of the crack front line:

$$Y_d = (\sqrt{\pi}MH_2) / \sqrt{Q} \quad (2)$$

where

$$Q = 1 + 1,464(a/c)^{1,65}$$

$$M = [1,13 - 0,09(a/c)]$$

$$+ \left\{ -0,54 + 0,89 \times [0,2 + (a/c)]^{-1} \right\} (a/W)^2$$

$$+ \left\{ 0,5 - [0,65 + (a/c)]^{-1} + 14 \times (1 - a/c)^{24} \right\} (a/W)^4$$

$$H_2 = 1 - [1,22 + 0,12(a/c)](a/W)$$

$$+ [0,55 - 1,05(a/c)^{0,75} + (a/c)^{1,5}] (a/W)^2$$

For the point at the surface of the crack front line:

$$Y_s = (\sqrt{\pi}MSH_1) / \sqrt{Q} \quad (3)$$

where

$$H_1 = 1 - [0,34 + 0,11(a/c)](a/W)$$

$$S = [1,1 + 0,35(a/W)^2] \sqrt{(a/c)}$$

EXAMPLE For $W = 3 \text{ mm}$ ($3 \times 10^{-3} \text{ m}$), $a = 50 \times 10^{-6} \text{ m}$, and $2c = 120 \times 10^{-6} \text{ m}$, $a/c = 0,833$, $a/W = 0,017$, $Y_d = 1,267$ and $Y_s = 1,292$.

9.2 Use the greater value of Y_d or Y_s for Y_{\max} and then compute the fracture toughness, K_{Isc} :

$$K_{\text{Isc}} = Y_{\max} \left[3PA/BW^2 \right] \sqrt{a} \quad (4)$$

where

K_{Isc} is the fracture toughness in megapascals by square root of a metre or meganewtons per metre to the power of 3/2;

Y_{\max} is the maximum stress intensity factor coefficient;

A is the four-point fixture moment arm in millimetres, $A = (S_o - S_i)/2$;

P is the break load in newtons;

B is the specimen width in millimetres;

W is the specimen depth in millimetres;

a is the crack depth in metres;

c is the crack half width in metres;

S_o is the outer (support) span in millimetres;

S_i is the inner (loading) span in millimetres.

NOTE The term in parentheses is the flexure strength of the precracked beam in megapascals. It is often useful to compare this value with the flexural strength of specimens without a precrack, in which fracture occurs from the natural fracture sources in the material.

9.3 If the specimens are chamfered, and if the chamfer sizes are larger than 0,15 mm, then fracture toughness values shall be corrected in accordance with Annex D.

9.4 If there is evidence of stable crack extension (8.3.7), then compute the fracture toughness, using Equation (4) and the critical crack size.

10 Test report

The test report shall include the following information:

- a) specimen identification;
- b) form of product tested (e.g. sintered, hot-pressed) if data are available;
- c) crack plane orientation if available;
- d) environment of test, relative humidity, temperature;
- e) specimen dimensions B and W ;
- f) crack dimensions, a and $2c$; a statement whether the maximum Y value was at the surface or at the deepest point of the crack periphery; a general description of the crack. If there is any evidence of stable crack extension, this evidence shall be reported as well as the initial precrack size and the critical crack size;

- g) fractographic equipment (optical or SEM) used and the approximate magnifications used to observe and measure the crack;
- h) method used to remove the indentation and residual stresses;
- i) displacement rate;
- j) number of valid test results;
- k) individual fracture toughness values, K_{Isc} ;
- l) mean fracture toughness and the standard deviation;
- m) whether there is evidence of stable crack extension. If yes, then a report of the fracture toughness based upon the critical crack size as well as the apparent toughness based upon the precrack size;
- n) whether the fracture toughness has been corrected for oversized chamfers;
- o) evidence of R-curve behaviour if detected;
- p) if tests have been performed at different loading rates, or different atmospheres, a report of the results for each testing condition.

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Annex A (informative)

Environmental effects

Some ceramics may be susceptible to slow crack growth phenomena caused by thermal or environmental factors. The measured values of fracture toughness may be a function of test rate and environment. Cracks may grow when under load and in an aggressive environment. The time-dependent phenomenon is known as “slow crack growth” (SCG). SCG can be significant even for short times during testing and can lead to measured fracture toughness values less than the inherent resistance in the absence of environmental effects.

Oxide ceramics, glasses and ceramics containing boundary phase glass are susceptible to SCG even at room temperature. Water, either in the form of liquid or as humidity in the air, can have a significant effect, even at the rates specified in this International Standard.

Time-dependent effects may be minimized through the use of an inert testing atmosphere, or the use of very fast loading rates. Testing specimens at two different loading rates may help determine whether the material is susceptible.

Even if testing is done in laboratory ambient conditions and the material is susceptible to slow crack growth, fractographic analysis may reveal the extent of the stable crack extension on the fracture surface. The stable extension sometimes may appear as a “halo” around the initial precrack as illustrated in Figures B.4, B.5 and B.7 d). The critical crack size at fracture, which is larger than the initial precrack size, should be used to calculate the environmentally-independent fracture toughness as discussed in References [18] and [19]. Halos are most commonly detected in fine-grained materials. It is difficult to detect them in medium- or coarse-grained ceramics.

Annex B (normative)

Precrack characterization

B.1 Techniques for crack characterization

B.1.1 Crack detectability varies considerably between ceramic materials. Since precracks are small, of the order 0,050 mm to 0,200 mm (50 μm to 200 μm) in size, fractographic methods are needed to find and characterize them. The detectability of cracks depends upon the material, the skill of a fractographer, the type of equipment used and the familiarity of the examiner with the material. It may be necessary to test ten specimens in order to obtain five specimens in which the cracks 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 is needed. The magnifications necessary for crack characterization are usually 100 \times to 500 \times . The superior depth of field of the scanning electron microscope is advantageous in many instances. Additional detail on detecting and measuring cracks is given in Reference [10].

B.1.2 Many ceramic materials have clear fractographic markings and the cracks are detectable with either optical or scanning electron microscopy. Examples are shown in Figures B.1 to B.4. In such cases, fracture toughness measurements on the same specimens using both optical and scanning electron microscopy crack measurements are in good agreement. The slight differences in size measurements have only a small influence on fracture toughness, due in a large part to the square root dependence of fracture toughness on crack size (References [3] and [5]).

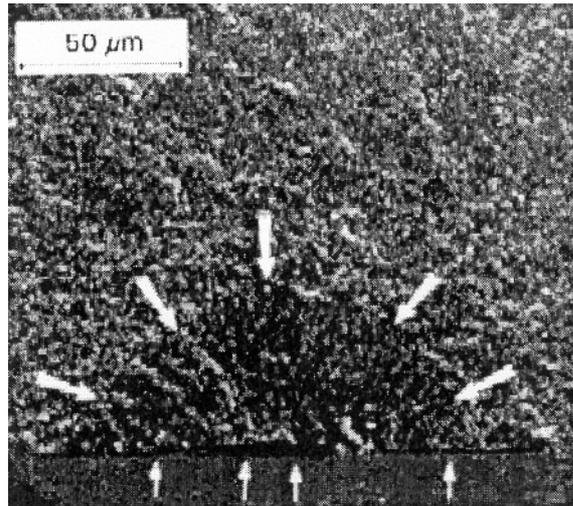
B.1.3 Many coarse-grained or incompletely-densified ceramics are not conducive to fractographic analysis. The surface crack in flexure method may not be suitable for these materials since no meaningful estimate of the crack size can be made. Larger indentation loads (≥ 98 N) may be helpful with medium- to coarse-grained materials. Larger precracks may stand out more clearly against the normal microstructural roughness on the fracture surfaces in such materials.

B.1.4 The precrack or critical crack may be detectable if:

- a) it is on a slightly different plane (angle) than the final fracture surface;
- b) it fractures in a different mode (transgranular) than the final fracture (intergranular);
- c) it leaves an arrest line;
- d) it has been dye penetrated or thermally tinted;
- e) it has coarse or fine hackle lines which change direction at the boundary.

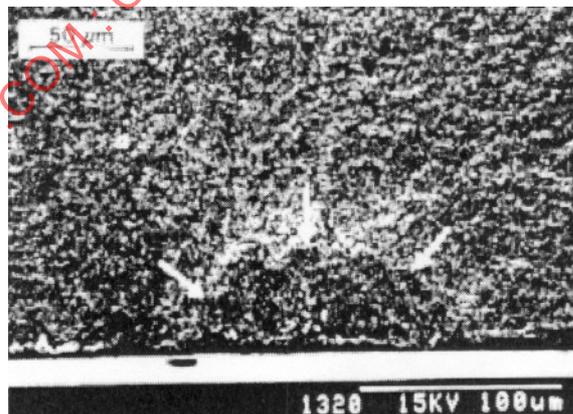
Conditions a) and b) will cause the crack to have a slightly different reflectivity or contrast than the rest of the fracture surface.

B.1.5 Dye penetration procedures may be helpful and are permitted by this International Standard, but a single simple procedure that works for all materials is not currently available. Dyes may be applied after indentation and prior to specimen fracture. Considerable caution should be exercised in the use of dye penetrants, since it is difficult to completely penetrate the small, tight cracks in ceramics. The optimum penetrant and impregnation procedure will vary between materials. Experience has shown that penetration procedures work best in "white" or light-coloured ceramics such as alumina and zirconia. Fluorescent penetrants may be needed for dark, opaque ceramics. Dye shall be dry prior to specimen fracture.



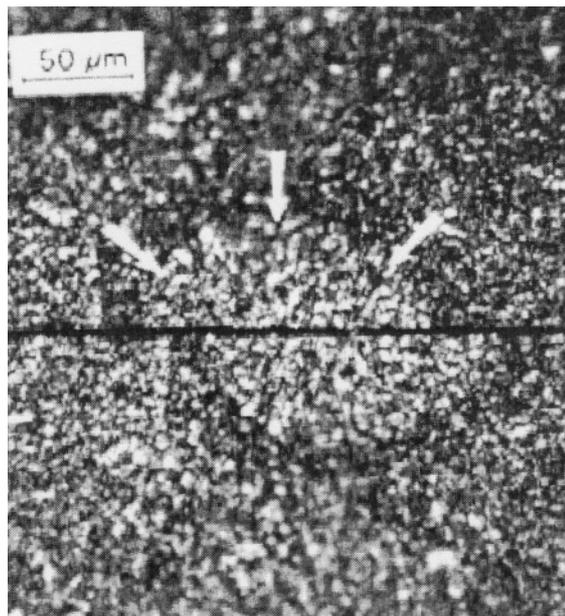
NOTE No material has been removed by polishing after indenting, and portions of the Knoop indentation are visible (small arrows).

Figure B.1 — Knoop indentation precrack in a hot-pressed silicon nitride as photographed by a scanning electron microscope



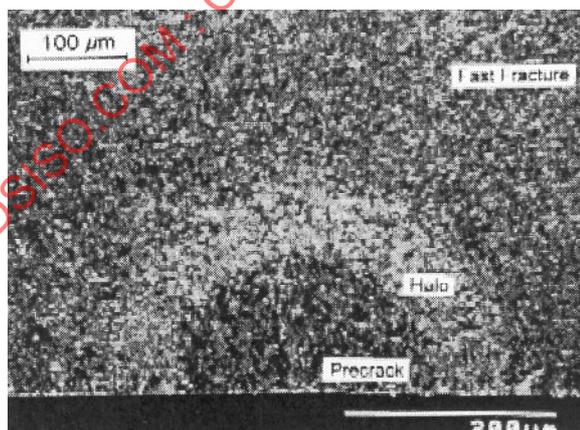
NOTE The slight “halo” at the top of the precrack is a “lip” due to crack realignment during specimen fracture.

Figure B.2 — Knoop indent crack in a hot-pressed silicon nitride as photographed by a scanning electron microscope



NOTE The fracture halves are mounted, back-to-back. The crack is the same as in Figure B.2.

Figure B.3 — Optical photomicrograph of a Knoop crack in hot-pressed silicon nitride



NOTE This specimen was tested in laboratory ambient conditions at room temperature. The white ring around the precrack is a "halo" of intergranularly-fractured material due to environmentally-assisted slow crack growth. Specimens of this same material tested in dry nitrogen do not have such "halos". The critical crack size includes the "halo".

Figure B.4 — Knoop indent crack in a 99,9 % fine-grained sintered alumina as photographed on the scanning electron microscope

B.1.6 Heat treatments may sometimes be useful in highlighting or “tinting” precracks, especially in some silicon carbides, but this approach is not permitted in this International Standard due to the risk of crack tip blunting or crack healing.

B.1.7 Both fracture surfaces shall be examined. The crack may be clearer on one than on the other.

B.1.8 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” or “lip” seen with either optical or electron microscopy if the precrack 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 Figure B.5. Sometimes such markings may be confused with stable crack extension, in which case interpretation can be difficult.

B.1.9 Fine hackle lines may change direction at a crack front line as shown in Figure B.6. Radiating hackle lines often give the precracks a fan-like appearance.

B.1.10 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×) photos often show the cracks quite clearly. At higher magnifications, contrast is lost in the optical or electron microscope or the depth of field is reduced in the optical microscope. A low power photo may be used to find and detect the crack and the higher power photo (100× to 500×) for measurements of the crack size.

B.1.11 Cracks often have subtle markings which cannot be discerned on scanning electron microscope (SEM) television monitors. Photography or a good digitally stored and printed image are essential with the SEM and will reveal cracks much better. Thermal prints should be used with caution, since considerable detail and clarity may be lost.

B.1.12 A strong specimen tilt (10° to 20°) during viewing may be effective with both optical and SEM microscopy. A photo can be taken which may show the crack quite clearly when tilted. Do not measure the crack size on such a photo since the crack dimensions will be foreshortened. A separate photo taken perpendicular to the fracture surface should be made and the two photos compared to help delineate and measure the crack.

B.1.13 Stereo photography with the SEM is extremely effective in showing crack topography and thus enabling a crack to be detected quite clearly, when it is almost undetectable by other means. Take one photo perpendicular to the crack, and a second photo at 10° to 20° off axis at the same magnification. A stereo viewer can be very helpful. Use the pair of photos to discern the crack, but take size measurements only from the photo that is perpendicular to the fracture surface.

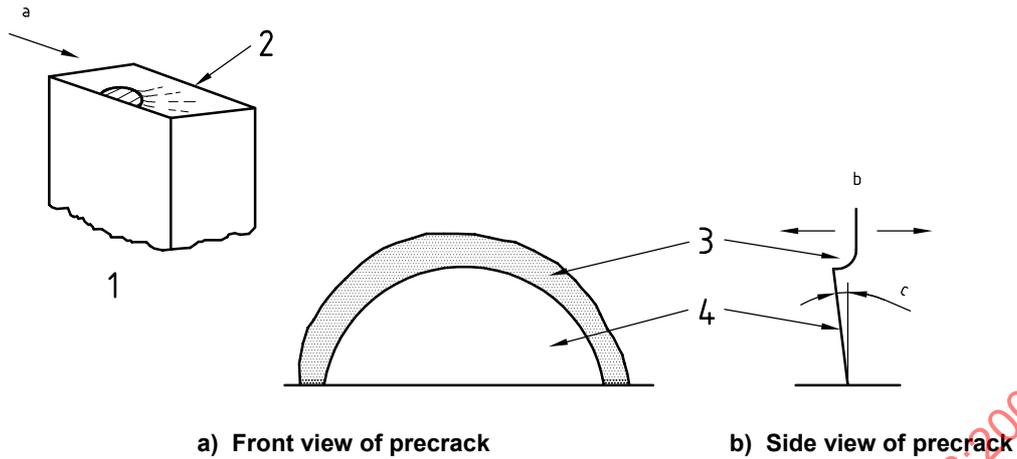
B.1.14 The back scattering mode in the SEM is helpful in some instances.

B.1.15 Thick gold-palladium coatings should not be used prior to scanning electron microscopy since such coatings can obscure fine detail. A 20 nm coating thickness is effective for most ceramics.

B.1.16 The gold-palladium coating may be applied at a strong angle (grazing incidence) to the fracture surface. This promotes contrast which will enhance fine detail.

B.1.17 A light gold-palladium coating may be very beneficial for optical microscopy on transparent or translucent “white” ceramics. The coating can mask internal reflections and light scatter.

B.1.18 A simple method of highlighting cracks in translucent or light-coloured ceramics after the specimen has been fractured is to “paint” the fracture surface with green ink from a common, office felt tip pen. Water or alcohol based office pens may be used. This technique is very effective when used with a stereo optical microscope. The fracture surface may be observed through the microscope while the green pen is applied to the fracture surface. The dye can easily be cleaned off with alcohol and the green ink painting process repeated.



Key

- 1 specimen
- 2 fracture surface
- 3 "halo"
- 4 precrack

NOTE The slight tilt of the precrack can create shadows or contrast differences when viewed in the optical or SEM. A uniform "halo" is indicative of environmentally-assisted slow crack growth. (See Figure B.4.) A "lip" at the precrack top is usually indicative of crack realignment during fracture. (See Figure B.2.)

- a Illumination direction.
- b Direction of applied stress, σ , during fracture.
- c Tilt.

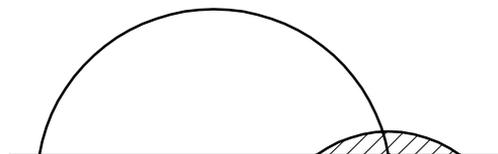
Figure B.5 — View of precrack and "halo" in the optical or SEM



Figure B.6 — Fine hackle lines which may change direction at the crack front line

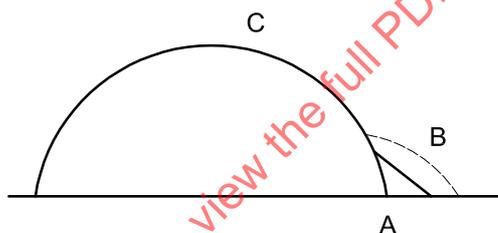
B.2 Crack characterization complications

Crack interpretation may be complicated by certain features on the fracture surface. Figures B.7 a) to f) provide guidance in such instances.



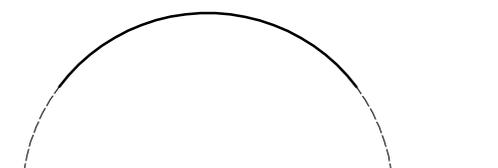
This can occur if the polishing or machining to remove the indent is too aggressive. Specimens with this damage can be repolished to remove the surface damage. If it is necessary to interpret such a crack, then approximate the semi-ellipse shape as if the surface damage were not present. If the maximum Y factor is at the surface, reject the datum (8.3.7).

a) Polishing or machining damage



During the fracture test, the crack reaches critical fracture condition at point A first. A small crack extends to B. Final fracture starts at point C. The original semi-ellipse should be used to compute fracture toughness. If the extension at points A-B is excessive, reject the datum. Polish or hand grind other specimens more in order to force the Y_{\max} to be at the deepest point, Y_d (8.3.8).

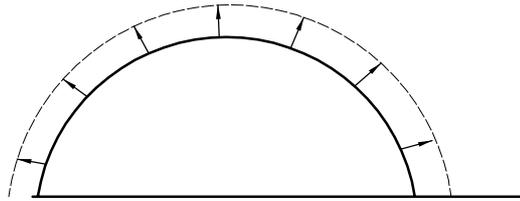
b) Corner pop-in



This can occur in instances where the crack and the final fracture crack are on the same plane. (The $1/2^\circ$ tilt may not have been adequate.) Alternatively, a limited depth of field in the optical microscope may hamper focusing the entire crack. Estimate or approximate the semi-ellipse shape as well as possible, but if more than 33 % of the crack is not visible, reject the datum (8.3.6).

c) Poorly defined crack at the surface

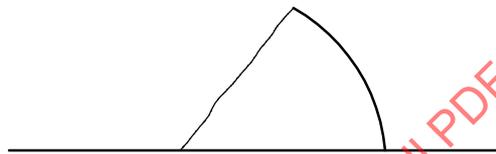
Figure B.7 — Crack interpretations



The crack may extend stably prior to fast fracture, either due to rising R-curve behaviour, 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 may be extremely difficult. If stable crack extension is detected, follow the guidelines in 8.3.9.

See Figure B.4 for an example of a crack with environmentally-assisted crack growth.

d) Stable crack extension



The final crack is on a different plane and only intersects 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 (8.3.6).

e) Precrack truncation



The precrack is actually made of three segments. The precrack is not flat and has a 3-dimensional aspect. 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 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 (8.3.6).

f) Precrack segmentation

Figure B.7 — Crack interpretations (continued)