INTERNATIONAL STANDARD

ISO 23146

Second edition 2012-12-15

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test methods for fracture toughness of monolithic ceramics — Single-edge V-notch beam (SEVNB) method

Céramiques techniques — Méthodes d'essai pour la détermination de la ténacité à la rupture des céramiques monolithiques — Méthode sur éprouvette à entaille en V sur une seule face (Méthode SEVNB)



Reference number ISO 23146:2012(E)

ISO 23146:2012(E)



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Published in Switzerland

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ISO 23146:2012(E)

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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 23146 was prepared by Technical Committee ISO/TC 206, Fine ceramics.

This second edition cancels and replaces the first edition (ISO 23146:2008), of which it constitutes a minor revision.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test methods for fracture toughness of monolithic ceramics — Single-edge V-notch beam (SEVNB) method

1 Scope

This International Standard specifies a method for the determination of the fracture toughness of advanced technical ceramics. The procedure makes use of single-edge V-notched bars, which are loaded in four-point bending until failure. It is applicable to monolithic ceramics with a grain size or major microstructural feature size larger than about $1~\mu m$.

The use of this International Standard for yttria tetragonal zirconia polycrystal material (Y-TZP) is not recommended. The method might also be unsuitable for some other very tough or soft ceramics in which a sharp crack does not form at the root of the V-notch.

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, Geometrical product specifications (GPS) — Dimensional measuring instruments; Micrometers for external measurements — Design and metrological characteristics

ISO7500-1, Metallic materials — Verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Verification and calibration of the force-measuring system

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

ISO 15732, Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for fracture toughness of monolithic ceramics at room temperature by single edge precracked beam (SEPB) method

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 15732 apply.

4 Principle

This method of conducting a fracture toughness test is based on the preparation and fracture of bar test pieces in which a sharp-tipped notch is machined. Using the technique of a reciprocating razor blade and diamond paste, a narrow notch can be honed into a test piece using either a manual method or a simple machine. Under well-controlled conditions, a notch-tip radius in the range of 1 μm to 20 μm can be prepared depending on the grain size of the test material. For many materials, this is a close approximation to a sharp crack, and the method has been found to give fracture toughness values very close to those of other methods such as the single-edge precracked beam method (ISO 15732) or the surface crack in flexure method (ISO 18756), provided that a sharp crack forms at the root of the notch either during its preparation or during the subsequent fracture sequence. The method has the advantage of simplicity of notch production compared with using a sharp-tipped diamond saw or a diamond impregnated wire in which the tip radius is normally greater than 50 μm . The method is often

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easier to undertake compared with other methods of precracking and is applicable to a wider range of materials outside the scope of these methods.

The method has been extensively researched (see the Bibliography) and has been evaluated in an ESIS (European Structural Integrity Society)/VAMAS (Versailles Agreement on Advanced Materials and Standards) round robin, the results of which are summarized in Annex B. This recommended practice is based upon the ESIS/VAMAS SEVNB round robin.

The method may have some limitations for materials with grain sizes of less than 1 μm , for which the assumption that the notch approximates to a sharp crack may not be valid (see Annex C). Users should strive to minimize the notch-root radius in the test pieces. This International Standard places a provisional criterion that the notch-root radius should be similar to or less than twice the average grain size of the material.

The method is complementary to other methods of measuring fracture toughness described in ISO 15732 (single-edge precracked beam method), ISO 18756 (surface crack in flexure method), and ISO 24370 (chevron notch method); see the Bibliography.

5 **Apparatus**

- **Ordinary razor blades**, preferably with a support along one edge but alternatively inserted in a suitable holder.
- Razor blades thinner than about 0,2 mm are not ideal for this task unless stiffened by a support, such as by gluing or screwing between two steel plates, leaving only about 2 mm of edge showing. A razor blade with a tip angle of 30° or smaller is ideal.
- Although it is possible to machine the notches entirely by hand, the use of a simple device in which the test pieces are mounted and in which a razor blade primed with diamond paste is moved in a reciprocating motion across the mounted test pieces has a distinct advantage, as this provides controlled loading and directional stability to the razor blade, and sharper notches are produced. An example is shown in Annex A.
- **Metallographic diamond paste**, in a viscous organic carrier and of fine grain size, typically 5.2 $1 \mu m$ to $3 \mu m$.
- 5.3 **Lubricant**, a light lubricating oil for lubricating the razor blade, e.g. clock oil.
- **Test piece support**, consisting of a flat plate or other suitable device for mounting test pieces 5.4 during notch honing.
- **Flexural-strength test fixture**, preferably a four-point flexural-strength test fixture operating in accordance with the requirements of ISO 14704, i.e. either 1/4-point flexure (preferred) or 1/3-point flexure. Alternatively, a three-point flexural-strength test jig may also be used, but the alignment of the V-notch with the central loading roller is more critical.

The test piece is supported on two bearing edges perpendicular to its length. The outer-support bearing edges shall be parallel rollers of diameter 5,0 mm \pm 0,2 mm and shall be capable of rolling outward on flat support surfaces. Preferably, one of the rollers shall additionally be capable of rotating about an axis parallel to the length of the test piece such that torsional loading is minimised. The two rollers shall be positioned initially with their centres $40.0 \text{ mm} \pm 0.5 \text{ mm} (1/4\text{-point flexure})$ or $30 \text{ mm} \pm 0.5 \text{ mm}$ (1/3-point flexure) apart with their axes parallel to within 1°. The separation of the centres of the rollers in their starting positions shall be measured to the nearest 0,1 mm with a travelling microscope. The rollers shall be made from hardened steel or other hard material with a hardness greater than 40 HRC (Rockwell C-scale). The rollers shall have a smooth burr-free surface finish with roughness less than $0.5 \, \mu m \, Ra$ and shall have a diameter uniform to $\pm 0.02 \, mm$.

For four-point flexure, the two loading rollers are located at the 1/4-points (or 1/3-points), with an inner span of 20 mm \pm 0,2 mm (or 10 mm \pm 0,2 mm for 1/3-point flexure) and are free to roll inwards. The rollers are free to rotate separately about an axis parallel to the length of the test piece to allow alignment. For three-point flexure, the single loading roller, which need not rotate, shall be positioned centrally between the outer-support rollers. The distances between the rollers shall be measured to the nearest 0,1 mm along the length of the specimen perpendicular to the direction of loading, using a travelling microscope or other suitable device. The loading rollers in four-point flexure shall be symmetrically positioned to within \pm 0,1 mm. The arrangement for loading shall ensure that equal forces are applied to the two loading rollers. The single loading roller in three-point flexure shall be centrally located to within \pm 0,2 mm.

5.6 Mechanical testing machine, capable of applying a force at a constant rate of displacement or constant loading rate to the test piece in the flexural-strength test jig and of recording the force at which the test piece fractures. The force measuring device shall be in accordance with ISO 7500-1 and shall have an accuracy of ≤ 1 %.

NOTE The test facility can, with advantage, be equipped with a capability for recording the force/displacement behaviour of the test piece, ideally a sensitive system directly contacting the test piece. Provided that the load-train stiffness is sufficient, machine displacement recording can be adequate.

- **5.7 Ultrasonic cleaning bath**, for cleaning the test pieces after notching, suitable for insertion of a beaker or other receptacle containing solvent.
- **5.8 Calibrated micrometer**, similar to the one in accordance with ISO 3611, but capable of being read to a precision of 0,002 mm using a vernier or electronic readout.
- **5.9 Optical microscope**, with calibrated magnifications over the range $50 \times to 500 \times$, suitable for observing the notch-tip shape, and fitted with photomicrographic facilities.
- **5.10 Notch-measuring device**, a calibrated device for measuring the depth of the sawn notch after fracture with a reading precision of 0,002 mm.

NOTE This can be achieved by the use of an appropriate travelling microscope, or a conventional metallurgical microscope with calibrated stage movement, or a microscope with a calibrated micrometer eyepiece.

- **5.11 Drying oven**, capable of maintaining 120 °C ± 5 °C for drying test pieces after cleaning.
- **5.12 Diamond slitting saw** or **slitting machine**, capable of preparing a shallow notch in a set of test pieces of width no more than 0,5 mm and depth of about 0,5 mm. See 6.3 for guidance.

6 Test piece preparation

6.1 Number of test pieces

At least seven test pieces shall be prepared for notching, of which five are required for testing and two are dummy test pieces for protecting the others during notch preparation.

NOTE If a machine is used for preparing the V-notches, it might not be necessary to employ the dummy test pieces to protect a test set of five test pieces.

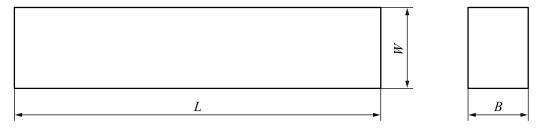
Operators with no experience of preparing the sharp V-notches used in this method are highly recommended to try out the technique and equipment first with surplus test pieces.

6.2 Test-piece dimensions

Prepare bar test pieces of rectangular cross-section, preferably in accordance with the requirements of ISO 14704. Figure 1 shows the shape and main dimensions of the test pieces prepared in accordance with this International Standard.

NOTE 1 The chamfering or rounding requirements in ISO 14704 are not essential for the V-notch test, and can be ignored.

NOTE 2 Other sizes of test pieces, e.g. (2 \times 2,5 \times > 25 mm), can optionally be used with appropriately sized flexural-strength fixtures.



Key

L > 45 mm (1/4-point flexure) or > 35 mm (1/3-point flexure)

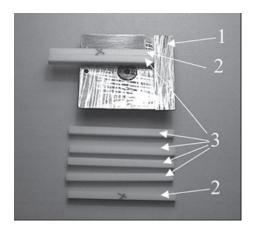
 $W = 4.0 \text{ mm} \pm 0.2 \text{ mm}$

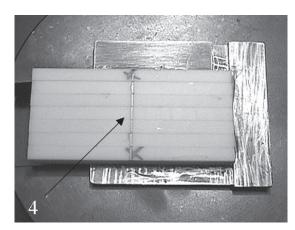
 $B = 3.0 \text{ mm} \pm 0.2 \text{ mm}$

Figure 1 — Test piece dimensions in accordance with ISO 14704

6.3 Preparing the V-notch by hand

Mount the test pieces side by side on the test-piece support using an appropriate temporary adhesive, as in Figure 2. Mount test pieces and dummies as close together as possible. Ensure that the top surfaces of the test pieces are level. Draw a pencil line across the set of test pieces at the mid-point of their length to indicate where the notch is to be prepared. Bending the test pieces while mounting on the holder is to be avoided.





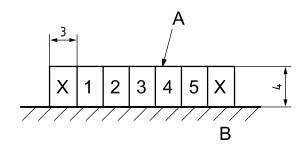
Key

- 1 mounting plate
- 2 dummy test pieces
- 3 test pieces for testing
- 4 pencil line as a guide for introducing a diamond-sawn starter notch

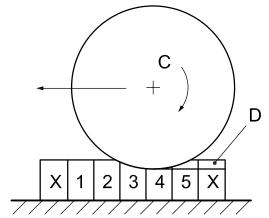
Figure 2 — Mounting procedure for the set of test pieces

Mount the holder on a diamond saw. Saw a starter notch along the pencil line of width ≤ 0.5 mm. The notch should have the same depth of about 0.5 mm over its entire length. Figure 3 shows a schematic arrangement for sawing. After sawing, clean the holder, test pieces, and especially the notch.

Dimensions in millimetres



a) Test pieces arranged on the test piece support



b) Machining a starter notch with a thin diamond saw blade

Key

- A tensile surface
- B holder
- C diamond saw blade
- D sawn notch
- X dummy test pieces

Figure 3 — Schematic diagrams for sawing

NOTE 1 It is helpful if the thickness of the diamond saw blade is only a little larger than the thickness of the razor blade used for notch honing. Otherwise, the razor-blade tip might skate over the surface of the pre-sawn notch and it might be difficult to start polishing the V-notch. V-shaping the saw-blade tip can help if available saw blades are significantly thicker than the razor blades.

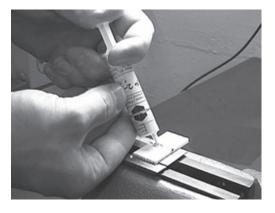
Fix the test-piece holder in a vice or other suitable clamp. Fill the starter notch with the fine diamond paste as shown in Figure 4 a).

To protect the fingers, place a razor blade in a holder or apply a heavy self-adhesive tape as shown in Figure 4 b). Put the exposed edge of the razor blade in the starter notch and apply a light force.

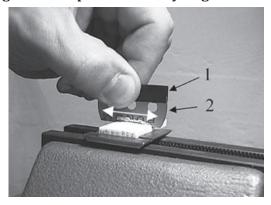
Excessive force can be counter-productive, causing the razor-blade tip to blunt more quickly. Using a force of about 5N to 10 N on the razor blade while polishing is normally sufficient.

Hone the V-notch with the razor blade with a smooth gentle back-and-forth motion [white arrows in Figure 4 b)]. Move the razor blade as upright and level as possible.

A drop of lubricating oil placed in the notch reduces any tendency for the razor blade to jerk or bind while honing.



a) Applying diamond paste from a syringe to the sawn notch



b) Reciprocating a razor blade in the sawn notch

Key

- heavy protection tape
- razor blade

Figure 4 — Positioning a razor blade in the sawn notch

Examine the depth of the V-notch periodically with an optical microscope at both ends of the V-notch. If possible, do not remove the test pieces from the test-piece holder for examination. If it is necessary to remove them and further honing is required, remount the test pieces on the holder with a razor blade inserted in the V-notch to act as a guide. If the total V-notch depth is less than 0,8 mm, continue to hone. The total final V-notch depth should be between 0,8 mm and 1,2 mm.

Honing by hand with a 4 µm diamond paste produces a V-notch width S of approximately 20 µm. Using a finer diamond paste, e.g. a 1 μ m one, usually does not decrease the width. If the V-notch width S is larger than about 30 μ m, it is suggested that a new razor blade or different razor-blade brand be employed. In coarse-grained materials, large grains might pop out into the notch during honing and limit the ability to obtain a sharp V-notch. NOTE 5 Different materials will take different periods of time to prepare the notch. Generally, harder or tougher ceramics take longer than softer, more easily machined ceramics. Polishing V-notches into alumina test pieces will take typically approximately 30 min and into silicon nitride ones typically approximately 4 h, for five test pieces and two dummies.

Remove the test pieces from the holder while avoiding bending them. Carefully clean the test pieces with acetone in a small powerful ultrasonic bath. Take care not to damage the test pieces while cleaning, e.g. by allowing them to come into contact during ultrasonic cleaning. Dry the test pieces in the oven for at least 1 h at $120\,^{\circ}\text{C}$.

NOTE 6 Cleaning of the V-notch can be difficult, especially when the tip radius is small. Alternative steps might be necessary to flush or scrape out the diamond grains.

6.4 Preparing the V-notches by machine (optional)

The task of honing V-notches can be automated. An example of a machine and its principal requirements are shown in Annex A.

NOTE 1 With a machine of sufficient rigidity employing a 1 μ m diamond paste for the final stages of notching, the notch width can be reduced typically to 1 μ m for ceramics with a grain size of about 1 μ m to 2 μ m.

NOTE 2 By commencing the notching directly on the flat test-piece surfaces using a razor blade and medium grit size diamond paste, typically $10~\mu m$, it is possible to avoid saw-notching the test pieces first. When the notch is deep enough, the notch tip is sharpened using a new blade and fine grit paste, after cleaning out the medium grit from the notch.

6.5 Determination of notch-root radius

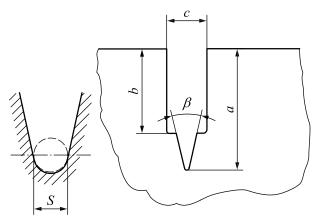
Select two test pieces from the set of five for testing. Photograph the V-notch on one side of each of the selected test pieces using a magnification of about $50\times$, or greater if the notch tip is not resolved at this magnification. Control the V-notch geometry with the help of the photograph. Report any deviation from the geometry shown in Figure 5.

Photograph the V-notch tip on the same two test pieces with a magnification approximately $300 \times .$ Measure the V-notch angle and width in accordance with Figure 5. Report the V-notch angle β and width S.

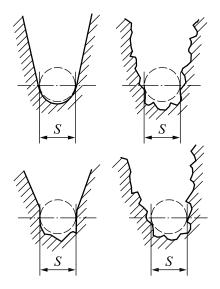
The notch width shall be of the same order as, or smaller than, twice the average grain size of the test material. Notches of width greater than 20 µm are not acceptable in any test material irrespective of grain size.

NOTE 1 Linear intercept methods for determining the average grain size are described in ASTM $E112^{[8]}$ or $EN 623-3^{[9]}$.

NOTE 2 It is helpful to determine the acceptability of the notch width on the outermost test pieces before removing them from the mounting plate. This allows the notch honing process to be continued if the notch width is too large.



a) Notch width and angle measurement



b) Interpretation of notch-root shape

Key

- *a* 0,8 up to 1,2 mm
- b approximately 0,5 mm
- c width of razor blade, a b > c
- β approximately 30° or as small as possible
- S V-notch width

Figure 5 — Schematic geometry of V-notches

7 Test procedure

7.1 Dimensions of test piece

Measure the depth W and width B (see Figure 1) of each test piece adjacent to the notch using the micrometer callipers. Read each value to the nearest 0,002 mm.

7.2 Flexural-strength test

Place the test piece in the test jig on its 3 mm width face, with the V-notched face resting on the outer-support rollers. Centralise the notch between the support rollers. If three-point flexure is employed, ensure that the notch is immediately under the loading roller to within ± 0.1 mm. Load the test piece such that the time to fracture is between 5 s and 15 s.

NOTE 1 This corresponds with a machine displacement rate of typically 0,5 mm/min.

Record the fracture load (maximum load) to three significant figures. The tests should be performed in air at room temperature. If subcritical crack growth is expected, the tests may also be conducted in a dry nitrogen atmosphere. Report the temperature and percentage relative humidity (% RH) during the tests or the medium in which the tests have been conducted.

Inspect the load/displacement curve for any evidence of subcritical crack growth or crack pop-in, particularly indicated by a downward curvature commencing just before fracture.

NOTE 2 The ability to detect subcritical crack growth can be strongly influenced by the test fixture and load-train stiffness. The use of test-piece-displacement measurement made directly on the test piece using a sensitive displacement transducer system, or a sensitive strain gauge placed on the test-piece surface opposite the notch, assists with detection.

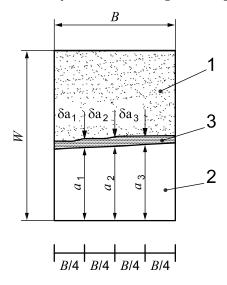
7.3 Measurement of notch depth

The depth of the V-notches is measured by observing the fractured surface (see Figure 6) using a microscope with calibrated stage movement and with a magnification $\geq 50 \times$. Read the depths a_1 , a_2 , and a_3 to three significant figures. Check if the fracture started at the bottom of the V-notch over its entire length; if not, the test is not valid.

NOTE 1 With white material, it might be difficult to measure the V-notch depth. In order to highlight the broken edges, typical techniques that can be used include grazing incidence illumination from a fibre-optic light-source or applying a coloured dye to the V-notch before fracture.

Inspect the region of fracture close to the notch tip at higher magnification and at angled or grazing incidence illumination. If evidence can be seen of a narrow zone (3 in Figure 6) where the crack has developed from the notch root or where this crack has propagated subcritically before fast fracture, determine the width of this zone, δa_1 , δa_2 , and δa_3 , in the same three positions as shown in Figure 6, and add these values to the notch lengths a_1 , a_2 , and a_3 , respectively.

NOTE 2 It might not be possible to see clearly the initial crack growth region in all test materials.



Key

- 1 fractured surface
- 2 V-notched surface
- 3 subcritical crack growth zone of width δa

Figure 6 — Schematic diagram of fractured V-notched test piece and the positions at which notch depths are measured

7.4 Calculation of fracture toughness

The average depth of the V-notch is calculated using Formula (1). The average relative V-notch depth α , calculated in accordance with Formula (3), shall be between 0,2 and 0,3 and shall satisfy Formula (2).

$$a = (a_1 + a_2 + a_3)/3 \tag{1}$$

$$\left(a_{\mathsf{max}} - a_{\mathsf{min}}\right) / a \le 0,1 \tag{2}$$

$$\alpha = a/W \tag{3}$$

For four-point flexure, compute the fracture toughness $K_{lc,SEVNB}$ using Formulae (4) and (5) (from ISO 15732):

$$K_{\text{lc,SEVNB}} = \frac{F}{B\sqrt{W}} \cdot \frac{S_1 - S_2}{W} \cdot \frac{3\sqrt{\alpha}}{2(1-\alpha)^{3/2}} \cdot Y^*$$
(4)

where

$$Y^* = 1,9887 - 1,326\alpha - \frac{(3,49 - 0,68\alpha + 1,35\alpha^2)\alpha(1-\alpha)}{(1+\alpha)^2}$$
 (5)

For three-point flexure using a 40 mm span, with 4 mm deep test pieces (i.e. $S_1/W = 10$), compute the fracture toughness $K_{\text{Ic.SEVNB}}$ using Formulae (6) and (7) (from ISO 15732):

$$K_{\text{lc,SEVNB}} = \frac{F}{B\sqrt{W}} \cdot \frac{S_1}{W} \cdot \frac{3\sqrt{\alpha}}{2(1-\alpha)^{3/2}} \cdot Y^*$$
(6)

where

$$Y^* = 1,9472 - 5,0247\alpha + 11,8954\alpha^2 - 18,0635\alpha^3 + 14,5986\alpha^4 - 4,6896\alpha^5$$
 (7)

For three-point flexure using a 30 mm span, with 4 mm deep test pieces (i.e. $S_1/W = 7.5$), compute the fracture toughness $K_{\text{Ic.SEVNB}}$ using Formulae (6) and (8) (from ISO 15732), where:

$$Y^* = 1,964 - 2,837\alpha + 13,7714\alpha^2 - 23,250\alpha^3 + 24,129\alpha^4$$
(8)

where

is the fracture toughness, in MPa • $m^{1/2}$; $K_{lc,SEVNB}$

is the fracture load, in MN;

is the test-piece width, in m; B

is the test-piece depth, in m; W

is the support span, in m; S_1

is the loading span ($S_1 > S_2$), in m; S_2

is the average V-notch depth, in m; a

is the maximum among a_1 , a_2 , a_3 , in m; $a_{\rm max}$

is the minimum among a_1 , a_2 , a_3 , in m; a_{\min}

is the relative V-notch depth; α

is the stress intensity shape factor.

The above calculations are considered valid for 0.35 < a/W < 0.6, and for the specified S_1/W values. ASTM C1421 (Reference [6] in the Bibliography) provides alternative equations for other S_1/W geometries.

Perform all calculations to three significant figures. Calculate the average $K_{lc,SEVNB}$ value and the standard deviation. Round the result to two decimal places.

NOTE 2 The four-point K_{lc} calculation can be checked with the following values: $F = 100 \times 10^{-6}$ MN, $B = 3 \times 10^{-3}$ m, $W = 4 \times 10^{-3}$ m, $a = 10^{-3}$ m, $S_1 = 40 \times 10^{-3}$ m, $S_2 = 20 \times 10^{-3}$ m. Result: $K_{\text{lc.SEVNB}} = 3,80$ MPa · m^{1/2}.

NOTE 3 The analysis above assumes that, at the point of fast fracture, the length of the crack developed from the notch tip is greater than the notch-root width *S*. If the crack length is significantly shorter than this, the fracture toughness value is overestimated. An additional correction factor is available (see Annex C).

8 Precision and bias

This method was evaluated in an international round-robin exercise on several materials. Details of the results of the round robin appear in Annex B. From these results, it would appear that, for materials with a grain size greater than about 1 μ m, the coefficient of variation of test results from test pieces in four-point flexure is typically less than 10 %, and the mean result is comparable with that achieved by other methods on similar materials. For materials with a finer grain size, the results appeared to continue to be dependent on the notch-tip radius and to give a higher result than the true result for a sharp crack.

This method assumes that the propagating crack initiates at the tip of the notch and that the notch length equals the sharp crack length. In practice, the sharp crack may have to initiate by linking honing damage at the tip of the notch, and the true critical crack length may be slightly longer than the measured notch length, leading to an underestimate of toughness of typically a few percent. The close observation of the crack surface close to the notch may permit identification of this subcritical initiation process and permit a correction to be made to the crack length measurements (see Figure 6 and Annex C). Subcritical effects are most likely to be observed with oxide materials tested in air of normal humidity.

9 Test report

The results shall be reported in accordance with the following minimum requirements:

- a) the name of the testing establishment;
- b) the date of receipt of the test pieces, the date of the test, a unique identification of the report and of each page, the name and address of the customer, and an authorized signatory of the report;
- c) a reference to this method, i.e. determined in accordance with ISO 23146:2012;
- d) identification of the test material type, manufacturing code, batch number, etc., and, where relevant, details of machining and any chamfering or rounding procedures employed to prepare test pieces;
- e) a description of the method employed to prepare the V-notches, the depth of the notches, and the notch-root width, including photographs;
- f) a description of the strength test jig, whether three-point or four-point flexure, and details of the fracture test procedure;
- g) measurements of the notch length after fracture and observations concerning obvious subcritical crack growth before fracture (either from compliance change or from fractographic evidence);
- h) the calculated fracture toughness for each test piece, the mean value of at least five valid tests, and the standard deviation;
- i) whether any adjustments to the fracture toughness calculations (see Annex C) were made for notchroot effects or stable crack extension;
- j) details of any necessary departure from the test conditions laid down in this International Standard as a result of the form or size of the test piece or the notch preparation method.

Annex A (informative)

Notch honing by machine

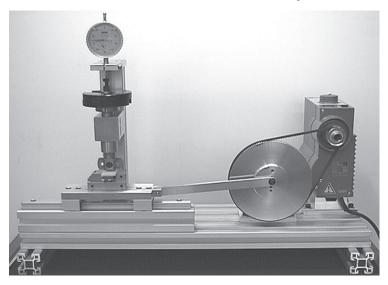
The process of notch honing is greatly simplified by employing a small machine with a reciprocating motion to either the razor blade or the set of test pieces. Figure A.1 shows an example of such a machine, in which a small electric motor drives a crankshaft attached to a linear slide on which the test pieces sit. The razor blade is clamped to the end of a vertical slide which has a small figure of eight applied. A dial gauge contacts the top of the slide shaft to give an indication of progress. It should be noted that the dial gauge gives the combined displacement of notch growth and blade wear. The notch growth needs to be checked by microscope. The alternative configuration is one in which the razor blade is reciprocated over stationary test pieces.

Whatever the configuration employed, the key features required of a machine are the following.

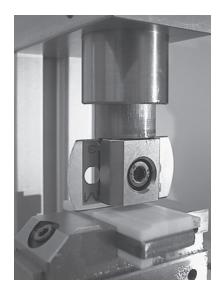
- The razor blade must be aligned accurately with the direction of motion of the machine; otherwise, it will bind in the notch, and its edge should be parallel to the direction of motion to ensure even wear and depth of honing.
- The razor blade must be replaceable with precise positioning in order to relocate in the sawn notch.
- The reciprocating motion must be adequately stiff laterally to avoid misalignments and binding.
- The test-piece mount should be precisely located on the machine base so that, if it is removed for inspection of test pieces, it can readily be replaced in the same position.

In operation, it will be found necessary to lubricate the blade and refresh the diamond paste typically every 10 min.

Using such a machine, it may not be necessary to provide a starter notch made by a diamond saw. It has been found that direct use of a razor blade can be made satisfactorily.



a) Simple reciprocating machine with moving test pieces



b) Details of the clamp for the test-piece mount and the razor blade

Figure~A.1-Simple~reciprocating~machine~with~moving~test~pieces~and~details~of~the~clamp~for~the~test-piece~mount~and~the~razor~blade

Annex B

(informative)

Interlaboratory evaluation of the SEVNB fracture toughness test procedures

In an international round robin under the auspices of ESIS and VAMAS with 31 participants, the fracture toughness was measured by the single-edge V-notched beam method in accordance with the procedure in this International Standard (see Reference [1] in the Bibliography). Details of the four monolithic advanced technical ceramics are listed in Table B.1. All of them possessed an average grain size or major microstructural feature size larger than 1 μ m.

Table B.1 — Processing parameters and material properties

Parameter	Alumina-998	Alumina-999	GPSSN	SSiC	
Powder	> 99,8 %	> 99,9 %	Bayer N3208		
Pressed sintered post HIPed	CIP ^a air	CIP ^a air gas-pressure	gas-pressure	HPb	
Grain size	> 10 µm	≈ 1,7 µm	< 1 µm, elonga- tion	7 μm	
Density	3,86 Mg/m ³	3,97 Mg/m ³	3,23 Mg/m ³	3,15 Mg/m ³	
Strength	342 MPa ^c	350 MPa ^c	> 920 MPa ^c		

a Cold isostatic pressed.

These ceramics had different degrees of difficulty in the application of this test method. Very consistent results were obtained for the alumina-998. The fracture toughness for the 135 valid tests in four-point flexure on size B test pieces from 28 participants was 3,57 \pm 0,22 MPa \cdot m¹/² (mean, standard deviation). Reasonably consistent results were obtained for the alumina-999. The fracture toughness for the 102 valid tests from 21 participants was 3,74 \pm 0,40 MPa \cdot m¹/². Consistent results were obtained for the GPSSN. The fracture toughness for the 129 valid tests accepted from 27 participants was 5,36 \pm 0,34 MPa \cdot m¹/² . Very consistent results were obtained for the SSiC. The fracture toughness for the 56 valid tests accepted from 12 participants was 2,61 \pm 0,18 MPa \cdot m¹/².

A smaller number of tests were also undertaken on yttria tetragonal zirconia polycrystal material (Y-TZP). These showed a clear evidence of notch-root radius dependence of outcome, and all outcomes gave higher values of toughness compared with the surface crack in flexure (SCF) method. None of the participants were able to achieve a sufficiently small notch-root radius. The method is therefore not recommended for this material.

To determine the repeatability (within-laboratory) and reproducibility (between-laboratories) of the SEVNB fracture toughness measurement method, ISO 5725-2 (Reference [2] in the Bibliography) was used in the round-robin analysis. The statistical results are shown in Table B.2 and are compared with results on the same materials from an earlier VAMAS round robin with the SCF method (Reference [3] in the Bibliography). The precision and bias statements for the SCF method were computed on the same basis but in accordance with ASTM E691 (Reference [4] in the Bibliography), in which repeatability and reproducibility are computed in an identical manner. (Note that in ISO 5725-2, the criterion for an outlier is 1 %, whereas in ASTM E691, it is 0,5 %.)

b Hot pressed.

c Four-point bending.

Details of the applicability of the method to small test pieces may be found in Reference [5] in the Bibliography.

Table B.2 — Repeatability and reproducibility coefficient of variance and standard deviation of the SEVNB method^[1] compared with the SCF method measured on hot-pressed Si_3N_4 [earlier VAMAS round robin (Reference [3] in the Bibliography)]

Material	Method	Total number of		Repeatability (within-labora- tory)		Reproducibility (between-laboratories)	
		participants	test pieces	Standard deviation	CV	Standard deviation	CV
				MPa⋅m ^{1/2}	%	MPa⋅m ^{1/2}	%
Alumina-998	SEVNB	28	135	0,17	4,6	0,22	6,1
Alumina-999	SEVNB	21	102	0,23	6,2	0,40	10,7
GPSSN	SEVNB	27	129	0,28	5,3	0,34	6,3
SSiC	SEVNB	12	56	0,12	4,5	0,18	6,8
Hot-pressed Si ₃ N ₄ Hot-iso-pressed Si ₃ N ₄	SCF SCF	19 15	102 100	0,24 0,38	5,4 7,7	0,31 0,45	6,8 8,9

Annex C

(informative)

Corrections for effective crack length greater than notch depth

The assumption in the mathematical analysis in this International Standard is that the effective crack length equals the notch depth. In practice, a sharp crack has to develop (i.e. grow subcritically or "popin") from the honing damage at the tip of the notch for fracture to occur, so that the true crack length at the peak force applied to the test piece is likely to be longer than that estimated from the notch depth, giving an underestimate of the true fracture toughness.

The presence of subcritical crack extension before fracture may be detected as a nonlinearity in the force/deflection curve of the test piece immediately before the peak force is attained. The extent of the subcritical extension of a crack-like flaw below the notch either has to be measured, e.g. from fractographic observation, or assumed from, for example, the grain size of the material, which may act as a flaw length limiter.

In order to provide an improved estimate of fracture toughness, an analysis given in Reference [6] in the Bibliography may be followed. Assuming that the test material shows no R-curve behaviour over the crack propagation distance between the notch root and the crack extension position at the point of fast fracture, Reference [6] shows that there is a negligible difference between true toughness and the toughness computed using the extended crack length (i.e. $a_0 + \delta a$) if $\delta a/R \ge 1.5$ for a planar crack or for a semi-elliptical crack extending from the notch tip, where δa is the length of the crack from the notch tip and *R* is the notch-root radius. In this condition, the nominal toughness computed with the notch depth can be corrected using the expression:

$$K^*_{\text{lc,SEVNB}} \approx K_{\text{lc,SEVNB}} \sqrt{1 + \delta a / a_0} \left\{ F \left[(a_0 + \delta a) / W \right] / F(a_0 / W) \right\}$$
(C.1)

where $F = Y^*$ in three-point flexure or $Y^*/(1 - \alpha)^{1,5}$ in four-point flexure. If the crack extension can be seen using fractographic methods and its length measured, this expression can validly be used when $\delta a/R \ge 1.5$.

If the condition is $\delta a/R < 1.5$, i.e. when the crack tip stress is strongly influenced by the radius of the notch, then there is an increasing error in using the above approach with decreasing δa or increasing R. An improved approximation is given by:

$$K^{**}_{lc,SEVNB} = K^{*}_{lc,SEVNB} \tanh(2,243\sqrt{\delta a/R})$$
 (C.2)

for a straight crack beneath the notch, or

$$K^{**}_{lc,SEVNB} = K^{*}_{lc,SEVNB} \tanh[2,243g(\delta a/R)\sqrt{\delta a/R}]$$
 (C.3)

for a semi-elliptical crack beneath the notch, where

$$g(\delta a/R) = 0.6667 + 0.178[1 - \exp(-1.64\delta a/R)]$$
 (C.4)

In cases where no obvious crack extension region can be identified fractographically, Damani et al. [10] suggest assuming that the extension should be accounted for, as being at least one grain size in length. For fine-grained materials, this may make a negligible correction, but for coarser grained materials, it may be very significant. The action taken by the user of this International Standard should be reported in the test report.

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