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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)

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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.

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.

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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 µm.

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

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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*

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ISO 7500-1, *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

5.1 Ordinary razor blades, preferably with a support along one edge but alternatively inserted in a suitable holder.

NOTE 1 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.

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NOTE 2 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.

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5.2 Metallographic diamond paste, in a viscous organic carrier and of fine grain size, typically 1 μm to 3 μm.

5.3 Lubricant, a light lubricating oil for lubricating the razor blade, e.g. clock oil.

5.4 Test piece support, consisting of a flat plate or other suitable device for mounting test pieces during notch honing.

5.5 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 ± 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 mm ± 0,5 mm (1/4-point flexure) or 30 mm ± 0,5 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 μm *Ra* and shall have a diameter uniform to ± 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 ± 0,2 mm (or 10 mm ± 0,2 mm for 1/3-point flexure) and are free to roll inwards. The

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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 ± 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 ± 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.

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5.9 Optical microscope, with calibrated magnifications over the range 50 × to 500 × , 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.

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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.

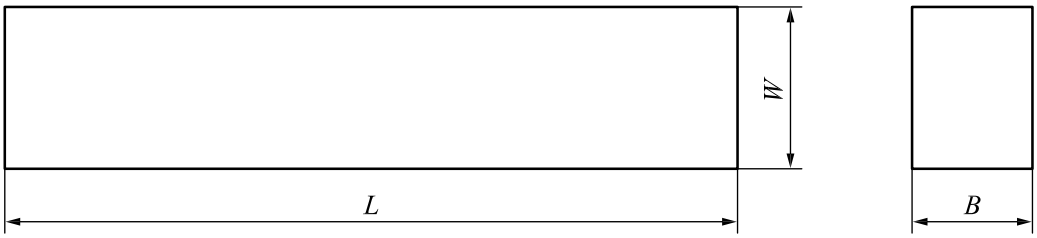
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6.2 Test-piece dimensions

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 × 2,5 × > 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 mm ± 0,2 mm
B 3,0 mm ± 0,2 mm

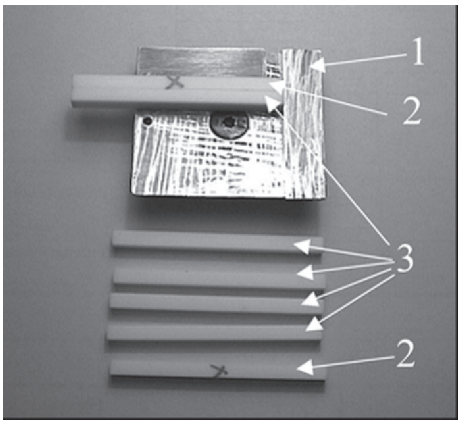
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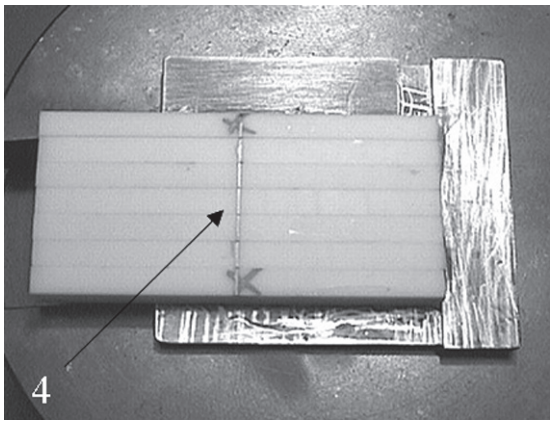
Figure 1 — Test piece dimensions in accordance with ISO 14704

6.3 Preparing the V-notch by hand

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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.





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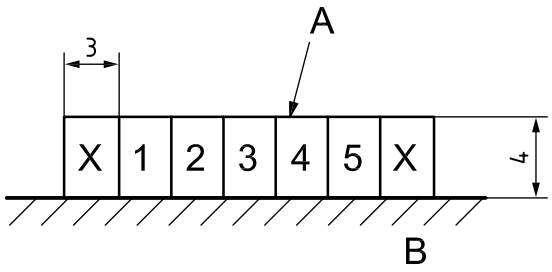
Key

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

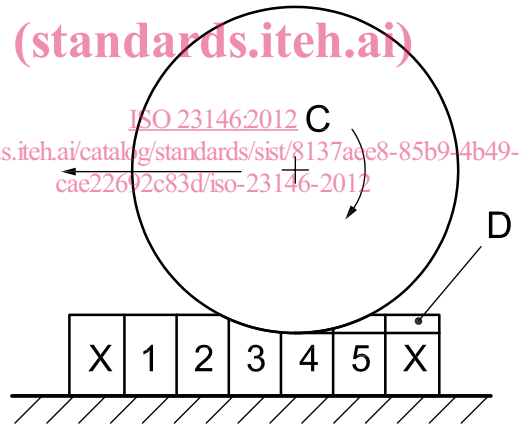
Mount the holder on a diamond saw. Saw a starter notch along the pencil line of width ≤ 0,5 mm. The notch should have the same depth of about 0,5 mm over its entire length. Figure 3 shows a schematic arrangement for sawing. After sawing, clean the holder, test pieces, and especially the notch.

Dimensions in millimetres



a) Test pieces arranged on the test piece support

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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).

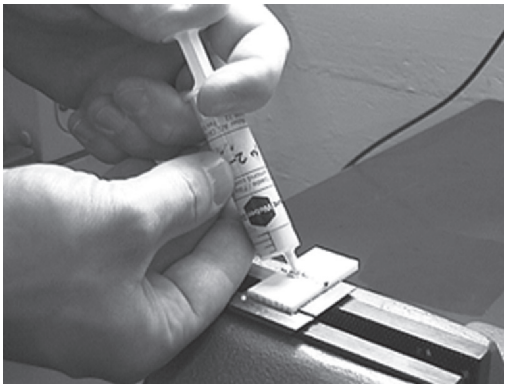
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To protect the fingers, place a razor blade in a holder or apply a heavy self-adhesive tape as shown in Figure 4 b). Put the exposed edge of the razor blade in the starter notch and apply a light force.

NOTE 2 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.

NOTE 3 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

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b) Reciprocating a razor blade in the sawn notch

Key

1 heavy protection tape
2 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.

NOTE 4 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 µm one, usually does not decrease the width. If the V-notch width S is larger than about 30 µm, 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.

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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 °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 µm diamond paste for the final stages of notching, the notch width can be reduced typically to 1 µm for ceramics with a grain size of about 1 µm to 2 µm.

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 µm, 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.

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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 × , 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.

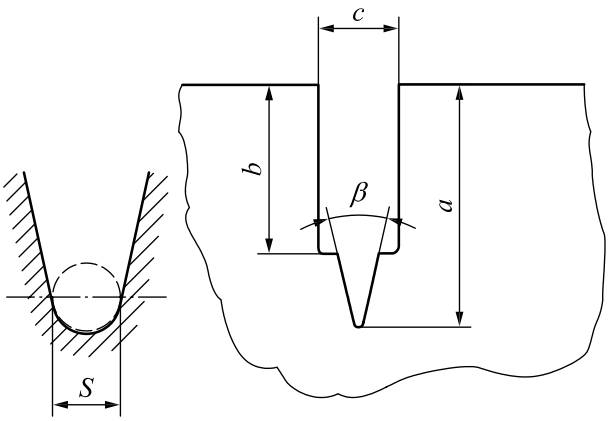
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Photograph the V-notch tip on the same two test pieces with a magnification approximately 300 × . Measure the V-notch angle and width in accordance with Figure 5. Report the V-notch angle β and width S.

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

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