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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for fracture toughness of monolithic ceramic thin plates at room temperature

iTeh STavancées) — Méthode d'essai pour déterminer la ténacité des plaques minces en céramique monolithique à température ambiante

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see <u>www.iso</u> .org/iso/foreword.html. (standards.iteh.ai)

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.isb/org/hiembers.html.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for fracture toughness of monolithic ceramic thin plates at room temperature

1 Scope

This document describes a test method for the determination of the fracture toughness of monolithic ceramic thin plates at room temperature by the single-edge precracked plate method or the single-edge V-notch plate method.

This document is intended for use with monolithic ceramics and whisker- or particulate-reinforced ceramics which are regarded as macroscopically homogeneous. It does not include continuous-fibre-reinforced ceramics composites. This document is applicable to ceramic thin plates with a thickness of 0,2 mm to <1,0 mm.

The single-edge V-notch plate method can be applied to monolithic ceramics with a grain size larger than about 1 μ m. The use of this method is inappropriate for yttria tetragonal zirconia polycrystal material (Y-TZP), which consists of homogeneous fine submicron grains. 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.

This document is for material development, material comparison, quality assurance, characterization, reliability and design data generation.

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2 Normative references rds.iteh.ai/catalog/standards/sist/37b65691-4db9-4232-

9cd7-406e18d2f5c3/iso-21113-2018 The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 1101, Geometrical Product Specifications (GPS) — Geometrical tolerancing — Tolerances of form, orientation, location and run-out

ISO 4287, Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters

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 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.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <u>https://www.iso.org/obp</u>
- IEC Electropedia: available at http://www.electropedia.org/

4 Principle

This method is to obtain the fracture toughness value from the precrack length or the V-notch depth, specimen dimensions and distance between the bending supports by measuring the fracture load of specimen according to the three- or four-point bending fracture test of a single-edge precracked plate specimen or a V-notched plate specimen. For the single-edge precracked plate method, a straight-through pop-in precrack is induced in the specimen via three-point bending a saw-notched test specimen bonded to a brass beam. For the V-notch plate method, a sharp-tipped notch is machined using the technique of a reciprocating razor blade and diamond paste.

The use of the single-edge V-notch plate method for materials with grain size less than 1 μm is not recommended, since the assumption that the notch approximates to a sharp crack is not established. Users should strive to minimize the notch-root radius in the test pieces. This document places a provisional criterion that the notch-root radius should be similar to or less than twice the average grain size of the material.

5 Apparatus

5.1 Bend testing machine.

A suitable testing machine capable of applying a uniform cross-head speed shall be used. The testing machine shall be in accordance with ISO 7500-1, with an accuracy of 1 % of the indicated force at fracture.

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5.2 Bend test fixture.

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The general features of the bend test fixture are illustrated in Figure 1. The bend test fixture shall be symmetrical about the centre line shown. The fixture shall have a thin slit in which the test specimen can stand vertically on the supporting roller pins. The width of the slit shall be about 0,03 mm larger than the specimen thickness. The loading, roller pin for the three-point bend test is placed in the positioning groove of the supporting member as shown in Figure 1 (a). The width of the positioning roller pins for the four-point bend test are placed in each positioning groove of the supporting member as shown in Figure 1 (b) and are held against the stops and pillars. For both three-point and four-point bend tests, the supporting roller pins are held against the stops and pillars. The loading roller pin from tilting so that it can push the specimen vertically. Other types of fixtures are acceptable; however, compression force shall be applied vertically on the top surface of the specimen. Materials composing the parts of the roller pins to be used shall be made of a material free from plastic deformation and risk of fracture. The diameter of loading roller pin and supporting roller pins shall be not more than 0,4 μ m.

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a) Three-point bend test fixture



b) Four-point bend test fixture

Кеу

- 1 loading ball
- 2 loading member with round head
- 3 loading member
- 4 tooth of the loading member
- 5 loading roller pins
- 6 specimen

- 7 precrack or V notch
- 8 supporting roller pins
- 9 stops and pillars
- 10 supporting member
- 11 longitudinal groove for the tooth of the loading member
- 12 slit for the specimen

Bending mode	Specimen	Diameter of roller pins mm	Support span d ₁ mm	Loading span d ₂ mm
3-point bend	Ι	4,0 to 5,0	30 ± 0,3	—
4-point bend	Ι	4,0 to 5,0	30 ± 0,3	10 ± 0,2
4-point bend	II	4,0 to 5,0	$40 \pm 0,4$	20 ± 0,2

Figure 1 — Bend test fixtures

6 Test piece dimension and preparation

6.1 Shape and dimensions of test piece

Prepare plate test pieces of rectangular cross-section. Figure 2 shows the shape and main dimensions of the test pieces. The parallelism tolerance on the opposite longitudinal side $L \times B$ face shall be 0,015 mm as defined in ISO 1101. The longitudinal side faces shall intersect with the large faces perpendicularly. The maximum variation in perpendicularity shall not exceed 0,01 mm. The variation of thickness, B_{max} - B_{min} , shall be less than 10 % of the average thickness, B_{ave} , which is determined from the thicknesses at three or more points.



Figure 2 — Dimensions of specimen

The longitudinal side $L \times B$ faces of as-cut thin plate usually intersect with the large face at a slightly oblique angle or have burrs and/or chippings as described in Figure 3. Both cross section and the longitudinal side $L \times B$ faces shall be observed with an optical microscope at a magnification of about 20 × or greater. The longitudinal side faces shall be polished until they form a right angle to the large surfaces.



Figure 3 — Guidelines for the unacceptable cross sections of specimen

It is recommended that the cut specimen width is slightly larger than 4,0 mm taking into account the reserving space for polishing. It is also recommended that the length, *L*, of the test piece is kept constant, for example 40 mm \pm 0,2 mm, unless special requests exist. Otherwise, different brass beams with stops need to be made for the ease of centring of the notch and precrack in the fixture every time the length of the test piece is changed.

6.2 Test piece preparation

Machining aspects unique to the precracked method and V-notched method shall be used as specified in <u>Annexes A</u> and <u>B</u>, respectively.

6.3 Number of test piece

At least eight test pieces shall be prepared for a flexural-strength test, of which five are required for valid results.

7 Test methods

7.1 Three or four-point bending fracture test

7.1.1 Insert the test piece into the slit of the test jig slowly, with the precracked or V-notched face on the outer-support rollers as shown in Figure 1. Ensure that the precrack is on the centre of supporting roller pins to within \pm 0,1 mm. Set the loading roller pin(s) on the specimen and hold the pin(s) until the loading member is placed on it, otherwise the loading roller pin(s) will drop off. When the loading member is positioned, turn the loading member so that the tooth fits in the longitudinal groove of the support member. If the tooth of the loading member is out of the longitudinal groove, the loading member and loading roller will tilt slightly, and compressive force will be applied obliquely to the test specimen.

7.1.2 Apply a compressive load to the loading member at a cross-head speed of 0,5 mm/min until the specimen fractures, and measure the maximum load, $P_{\rm f}$ to an accuracy of 1 %.

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7.2 Measurement of thickness and width of test piece

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The measurement of the thickness and width of the specimen shall be carried out using a micrometer or other device accurate to the nearest 0.01 mm 3/iso-21113-2018

7.3 Measurement of precrack length

For the single-edge precracked plate method, measure the precrack length of the test piece after fracture as specified in <u>Annex C</u>.

8 Evaluation of validity of measured value

A valid test shall meet the requirements specified in <u>Annexes B</u> and <u>C</u>.

9 Calculation

Calculate the fracture toughness value, K_{Ipp} or $K_{Ic,SEVNP}$, from the measured values for each specimen according to Formulae (1) to (4). Obtain the average value of fracture toughness of five or more specimens conforming to the specification given in Clause 8 and round off to one decimal place.

$$K_{\rm Ipp} \, or \, K_{\rm Ic,SEVNP} = \frac{P_{\rm f} \times d_1}{B \times W^{3/2}} \left[\frac{3}{2} \alpha^{1/2} \times Y(\alpha) \right] \tag{1}$$

where, in the case of three-point bend specimen I,

$$Y(\alpha) = 1,964 - 2,837 \alpha + 13,711 \alpha^2 - 23,250 \alpha^3 + 24,129 \alpha^4$$
(2)

In the case of four-point bend specimen I and II,

$$K_{\rm Ipp} \, or \, K_{\rm Ic,SEVNP} = \frac{P_{\rm f} \times (d_1 - d_2)}{B \times W^{3/2}} \left[\frac{3}{2} \frac{\alpha^{1/2}}{(1 - \alpha)^{3/2}} \times Y(\alpha) \right]$$
(3)

$$Y(\alpha) = 1,987\ 7 - 1,326\ \alpha - \frac{(3,49 - 0,68\ \alpha + 1,35\ \alpha^2)\alpha(1 - \alpha)}{(1 + \alpha)^2}$$
(4)

where

 K_{Ipp} is the fracture toughness, single-edge precracked plate method, in MPa·m^{1/2};

 $K_{\text{Ic,SEVNP}}$ is the fracture toughness, single edge V notch plate method, in MPa·m^{1/2};

- $P_{\rm f}$ is the fracture load, in MN; <u>ISO 21113:2018</u>
- *B* is the specimen thickness, in m, standards/sist/37b65691-4db9-4232-9cd7-406e18d2f5c3/iso-21113-2018
- W is the specimen width, in m;
- d_1 is the support span, in m;
- d_2 is the loading span ($d_1 > d_2$), in m;
- α is the average relative precrack length or the average relative V-notch depth;

Y is the stress intensity shape factor.

The mean fracture toughness, K_{Ipp} or $K_{Ic,SEVNP}$, and the standard deviation, *s*, are given by Formulae (5) and (6), respectively.

$$\overline{K}_{Ipp} = \frac{\sum_{i=1}^{n} K_{Ipp,i}}{n} \text{ or } \overline{K}_{Ic,SEVNP} = \frac{\sum_{i=1}^{n} K_{Ic,SEVNP,i}}{n}$$
(5)
$$s = \left[\frac{\sum_{i=1}^{n} (K_{Ipp,i} - \overline{K}_{Ipp})^{2}}{n-1}\right]^{1/2} \text{ or } s = \left[\frac{\sum_{i=1}^{n} (K_{Ic,SEVNP,i} - \overline{K}_{Ic,SEVNP})^{2}}{n-1}\right]^{1/2}$$
(6)

where

*K*_{Ipp,*i*} is the fracture toughness of the *i*th specimen, single-edge precracked plate method;

*K*_{Ic,SEVNP,*i*} is the fracture toughness of the *i*th specimen, single-edge V-notch plate method;

n is the total number of specimens.

10 Test report

The test report shall include the following information: **PREVIEW**

- a) type of material, code, batch number, date of manufacture;
- b) type of test method (either single-edge precrack plate method or single-edge V-notch plate method);
- c) number of specimens conforming to the specifications given in <u>Clause 8</u>;
- d) measured fracture toughness values, K_{Ipp} or $K_{\text{Ic,SEVNP}}$, the average and the standard deviation, in megapascal metres to the power half;
- e) specimen dimensions: thickness, *B*, width, *W*, in millimetres;
- f) precracking conditions (e.g. depth and width of notch and pop-in load) or a description of the method employed to prepare the V-notches;
- g) environment conditions, relative humidity and temperature;
- h) maximum load before fracture, *P*_f, in newtons;
- i) precrack lengths, *l*, in millimetres, or depth of the V-notches, *a*, notch-root width, *S*, in millimetres, and V-notch angle, β .