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Second edition
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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for fracture resistance of monolithic ceramics at room temperature by indentation fracture (IF) method

Céramiques techniques (céramiques avancées, céramiques techniques avancées) — Méthode d'essai pour déterminer la résistance à la rupture par indentation (IF) des céramiques monolithiques à température ambiante

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Foreword

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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 document 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).

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

This second edition cancels and replaces the first edition (ISO 21618:2019), of which it constitutes a minor revision. The changes are as follows:

— replacement of the withdrawn normative reference ISO 4287 by the current equivalent standard ISO 21920-2:
— editorial changes.

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.iso.org/members.html.

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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for fracture resistance of monolithic ceramics at room temperature by indentation fracture (IF) method

1 Scope

This document specifies a test method for determination of the fracture resistance of monolithic ceramics at room temperature using the indentation fracture (IF) method.

This document is applicable to dense monolithic ceramics and whisker- or particulate-reinforced ceramics which are regarded as macroscopically homogeneous. This document is not applicable to monolithic silicon nitride ceramics for bearing balls and continuous-fibre-reinforced ceramics composites.

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

Indentation fracture resistance, $K_{I,IFR}$, as defined in this document is not to be equated with fracture toughness determined using other test methods such as K_{Isc} and K_{Ipb}.

NOTE $K_{I,IFR}$ is an estimate of a material's resistance to cracking as introduced by an indenter and has correlations with wear resistance and rolling contact fatigue performance as well as machining processes, since these properties are governed by the resistance to crack extension in localized damage areas.[1]-[3] By contrast, fracture toughness properties K_{Isc} and K_{Ipb} are intrinsic properties of a material and are relevant to macroscopic and catastrophic fracture events with long cracks rather than those phenomena caused by microscopic and successive damage accumulation associated with short cracks.

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2 Normative references

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 6507-2, *Metallic materials — Vickers hardness test — Part 2: Verification and calibration of testing machines*

ISO 6507-3, *Metallic materials — Vickers hardness test — Part 3: Calibration of reference blocks*

ISO 14705, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for hardness of monolithic ceramics at room temperature*

ISO 17561, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for elastic moduli of monolithic ceramics at room temperature by sonic resonance*

ISO 21920-2, *Geometrical product specifications (GPS) — Surface texture: Profile — Part 2: Terms, definitions and surface texture parameters*

3 Terms and definitions

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

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

— ISO Online browsing platform: available at https://www.iso.org/obp

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— IEC Electropedia: available at https://www.electropedia.org/

3.1
fracture resistance
measure of resistance of extension of a crack introduced by an indenter

3.2
fracture resistance value
$K_{I,IFR}$
value of crack-extension resistance as measured by the indentation fracture (IF) method

Note 1 to entry: The indentation fracture resistance, $K_{I,IFR}$, as used here is not to be equated to fast fracture toughness K_{Ic}. $K_{I,IFR}$ is an estimate of a material's resistance to cracking as introduced by an indenter. K_{Ic} is considered to be an intrinsic property of a material and is independent of the test method.

4 Principle

This document is for material development, material comparison, quality assurance, characterization and reliability data generation of dense monolithic ceramics. The method determines the indentation fracture resistance value, $K_{I,IFR}$, from the elastic modulus and indentation force by forcing a Vickers indenter (diamond pyramid) into the surface of a test piece and measuring the lengths of both the diagonals and the associated cracks of the indentation that are left in the surface after removal of the indenter (see Figure 1). [4] The method is applicable to the half-penny-crack type but not to the Palmqvist type. The two types of crack profile can be estimated using the ratio of the crack length, $2c$, to the diagonal length, $2a$. If $2c/2a$ is more than 2,5, the crack is half-penny type. But the critical $2c/2a$ values can be smaller than 2,5 for some ceramics. In such cases, direct observation of crack morphology by the decoration technique or serial sectioning of the indented surface is also useful to differentiate the two crack profiles. The measurement of the crack length and the observation of the crack tips are performed separately in order to improve the accuracy. The crack length should not be measured in a single optical image because doing so inevitably limits the magnification that can be used. Travelling microscopy is a solution that allows both reading of the crack length and detection of the crack tips to be performed at high resolution, albeit separately. Both an objective lens of 40 × or higher and a calibrated optical stage shall be employed to ensure reliability. Both international and domestic interlaboratory comparison study (round robin) projects on the advanced IF method are described in Annex C (see References [5] to [8]).

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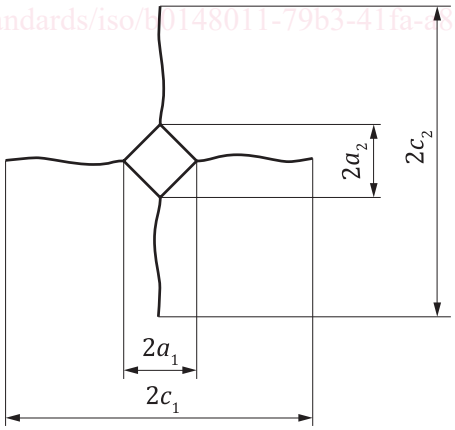

Key
$2a$ diagonal length of the indent
$2c$ diagonal length of the crack

Figure 1 — Crack lengths and diagonal sizes of the Vickers indentation

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5 Apparatus

5.1 Testing machine

The testing machine shall be in accordance with ISO 6507-2. A suitable testing machine capable of applying an indentation force of 49,03 N up to 196,1 N shall be used.

5.2 Indenter

The indenter shall meet the specification for Vickers indenters. See test method ISO 6507-2. The diamond should be examined before the test, and if it is loose in the mounting material, chipped or cracked it shall be replaced.

5.3 Verification by standard reference materials

Reference materials which are in accordance with ISO 6507-3 shall be used to verify the testing machine and their Vickers hardness shall not vary from the hardness of the material to be measured by more than 20 %.

5.4 Metallurgical microscope or travelling microscope

The metallurgical microscope equipped with calibrated stage movement or the travelling microscope shall be employed for both detection of the crack tips and measurement of the crack length. The magnification of the objective lens shall be 40 × or 50 × and the total magnification shall be 400 × or more. The travelling stage shall move both vertically and horizontally and the readout resolution of the table position shall be 1 µm or less. The coaxial vertical illumination with enough light intensity shall be used for the observation of the crack tips.

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6 Test specimen

6.1 Thickness

The thickness of the specimen shall be large enough so that the crack lengths are not affected by variations in the thickness. As long as the thickness of the specimen is more than five times the crack length (2*c*), the test will not be affected. In general, a specimen thickness of more than 3 mm is suitable.

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6.2 Surface finish

Specimens shall have a ground and polished surface so that the crack lengths can be measured accurately. The surface roughness, *Ra*, as defined in ISO 21920-2, shall be not more than 0,1 µm. Any grinding-induced damage layer at the surface shall be removed completely by polishing so that the crack lengths are not affected by any residual stress at the surface layer. The area fraction of tiny pits due to dropout of a grain or fragmentation shall be as small as possible to enable the precious crack-length measurement.

NOTE Annex A provides further information about a typical machining procedure.

The IF method is only applicable to dense ceramics. But if it is necessary to evaluate slightly porous ceramics through the IF method, the porosity shall be described in the test report to show the precision of measurements, because ceramics with some small porosity can experience local densification beneath the indenter, which reduces the driving force for crack formation and results in overestimation of $K_{\mathrm{I,IFR}}$.

7 Procedure

7.1 Specimen placement

Place the specimen on the stage of the machine so that the specimen will not rock or shift during the measurement. The specimen surface shall be clean and free from any grease or film.

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7.2 Specimen levelling

The surface of the specimen being tested shall lie in a plane normal to the axis of the indenter.

7.3 Cleaning of the indenter

The indenter shall be cleaned prior to and during a test series. A cotton swab with ethanol, methanol or isopropanol may be used. Indenting into soft copper can also help remove debris.

7.4 Adjustment of stage movement

Ensure that the horizontal movement of the stage of optics is coincident with the horizontal direction in the field of optics. The magnitude of the stage movement shall be calibrated with an objective micrometer. The procedures for the adjustment of the stage axis and for the length calibration shall be carried out as specified in Annex B.

7.5 Application of test force

Indentations shall be made using a Vickers indenter under the following conditions:

— Force: 196,1 N;

— Dwell time: 15 s.

If indentations made at the test force of 196,1 N lead to no acceptable indentations (see Figure 2), use a lower test force of 98,07 N or 49,03 N. The test force employed shall be described in the test report to show the precision of measurements.

NOTE $K_{I,IFR}$ measured at a force of 98,07 N or 49,03 N can be slightly smaller than that obtained at 196,1 N, especially for those silicon nitrides with self-reinforced microstructures which produce rising R-curve behaviour. Also, the accuracy of the measurement of crack length can become worse when the indentation size gets smaller at the test force of 98,07 N or 49,03 N.

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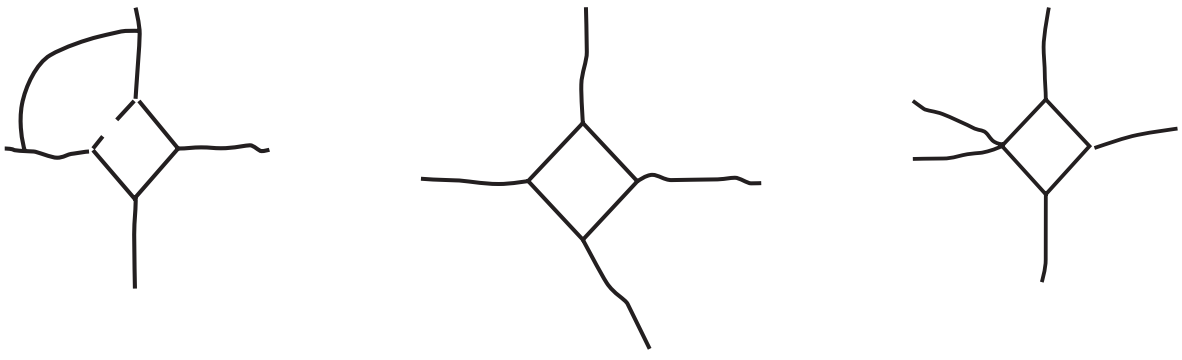
7.6 Acceptability of indentations

Only indentations whose four primary cracks emanate straight and radially from each corner shall be accepted. Indentations with spalled edges, badly asymmetrical, split or forked cracks or gross chipping shall be rejected, see Figure 2. If the difference between the horizontal crack length and the vertical length is more than 10 % of the mean value of the horizontal and vertical lengths, the result shall be rejected. If $2c/2a$ is less than 2,5, the results shall be rejected since the crack can be the Palmqvist type. However, if the crack type is half-penny, the results are acceptable. The crack type can be differentiated by decoration of cracks using impregnation of lead acetate solution or by the serial sectioning technique.[9] The number of both valid indentations and total indentations shall be described in the test report to show the precision of measurements, since a large percentage of invalid indentations implies measurements using valid indentations can be affected somewhat by, for example, the potential lateral cracks.



a) spalled edge **b) asymmetrical** **c) badly split**

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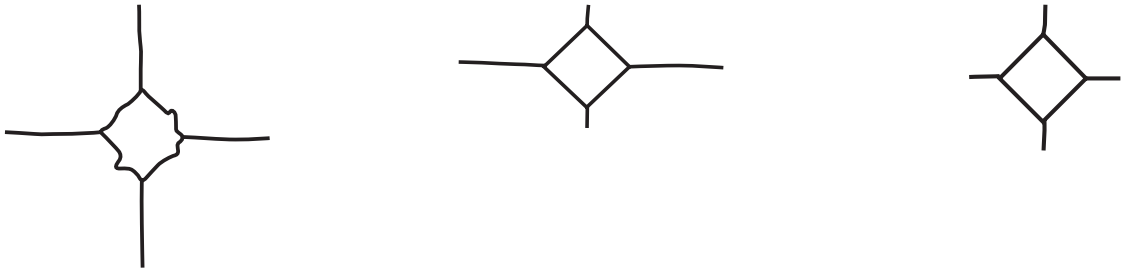
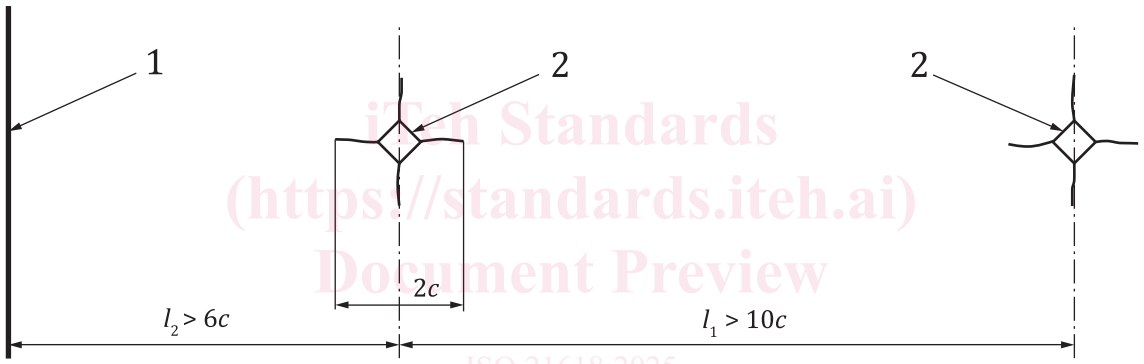


d) chipping and ragged edges **e) $2c_2 << 2c_1$ or $2c_1 << 2c_2$** **f) $2c/2a < 2{,}5$**

Figure 2 — Guidelines for the unacceptable indentations

7.7 Number of indentations

The number of valid indentations shall be not less than five. The spacing of indentations shall not be less than five times the diagonal length of cracks. See Figure 3. No indentation shall be closer than three times the diagonal length of the crack to the test-piece edge.



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Key

1 edge of test piece
2 indentations
$2c$ diagonal length of the crack
l_1 distance between centres of indentations
l_2 distance from the centre of indentation to the edge of the sample

Figure 3 — Closest permitted spacing between indentations and from indentation to the test piece edge for Vickers indentations

7.8 Measurement of indentation size

7.8.1 Measure both diagonals of each hardness impression as "$2a$" values to within 1 µm within 10 min of unloading. The measurement procedure specified in ISO 14705 or the following apply.

7.8.2 Adjust the diagonal directions to both vertical and horizontal axes of the field of optics.

7.8.3 Centre the left corner of the diagonal in the field of optics and record the stage position.

7.8.4 Shift the stage to centre the right corner of the diagonal in the field of optics and record the stage position.

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7.8.5 Calculate the horizontal diagonal size, $2a_1$, from the change of stage position.

7.8.6 Obtain the vertical diagonal size, $2a_2$, following the same procedure as 7.8.3 to 7.8.5.

7.9 Measurement of crack size

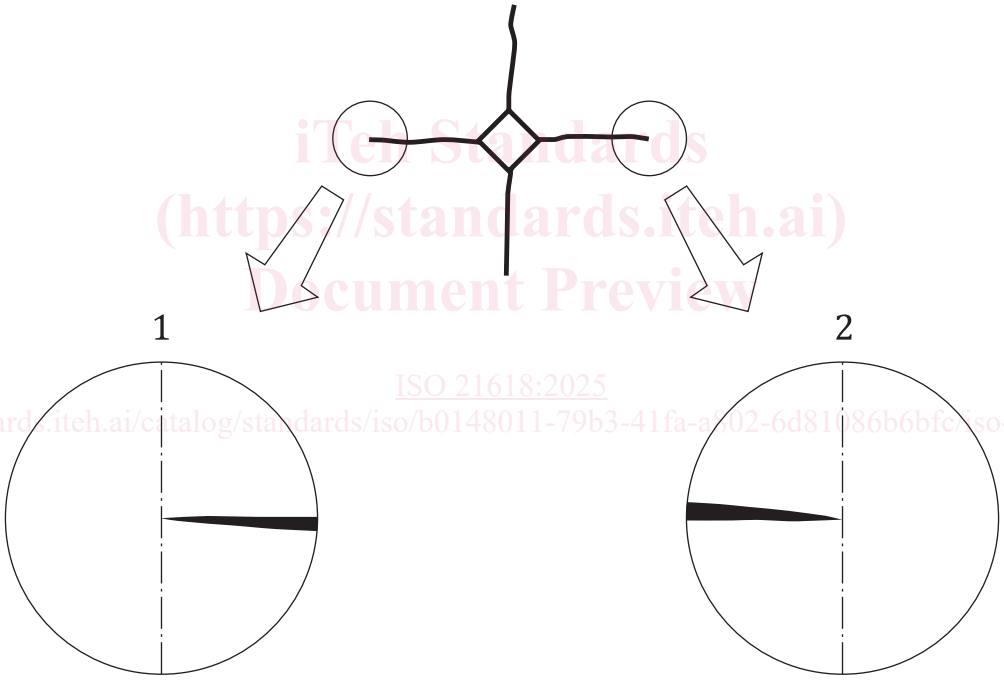
7.9.1 Measure both horizontal and vertical crack length as "$2c$" values to within 1 µm within 10 min of unloading. The following measurement procedure shall be used.

7.9.2 Centre the left crack tip in the field of optics as shown in Figure 4 (left) and record the stage position. To identify the crack-tip position, it is recommended that the image is brought into focus and out of focus several times by turning the fine knob back and forward quickly.

7.9.3 Shift the stage to centre the right crack tip in the field of optics as shown in Figure 4 (right) and record the stage position.

7.9.4 Calculate the horizontal crack length, $2c_1$, from the change of stage position.

7.9.5 Obtain the vertical crack length, $2c_2$, following the same procedure as 7.9.2 to 7.9.4.



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Key

1 observation of the left crack tip at a high magnification (the left crack tip is centred in the field of optics)

2 observation of the right crack tip at a high magnification (the right crack tip is centred in the field of optics)

Figure 4 — Measurement method for the crack length using both objective lens of 40 × or 50 × and travelling stage

7.9.6 If the crack tip touches a void with a diameter of 10 µm or more, the data shall be rejected since it is difficult to locate the precise position of the crack tip. If the diameter of the void is less than 10 µm, measure the crack length including the void diameter, as shown in Figure 5 (left). When the crack is interrupted at a grain or void and appears again, take the end of the disconnected crack as the crack tip as shown in Figure 5 (right) and measure the crack length.

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