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

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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 describes a test method that covers the determination of fracture resistance of monolithic ceramics at room temperature by the indentation fracture (IF) method.

This document is intended for use with dense monolithic ceramics and whisker- or particulate-reinforced ceramics which are regarded as macroscopically homogeneous. It does not include monolithic silicon nitride ceramics for bearing balls and continuous-fibre-reinforced ceramics composites.

This document is 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.

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

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*

3 Terms and definitions

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

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

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

— IEC Electropedia: available at http://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 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]).
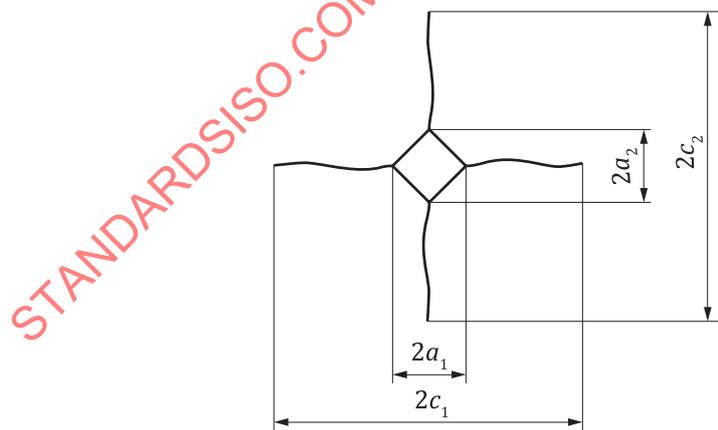
Key

$2a$ diagonal length of the indent

$2c$ diagonal length of the crack

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

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

The 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.

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 ($2c$), the test will not be affected. In general, a specimen thickness of more than 3 mm is suitable.

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 4287, 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 $K_{I,IFR}$.

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.

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 $K_{\text{I,IFR}}$ 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.

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 could 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.

Key

1 spalled edge
2 asymmetrical
3 badly split
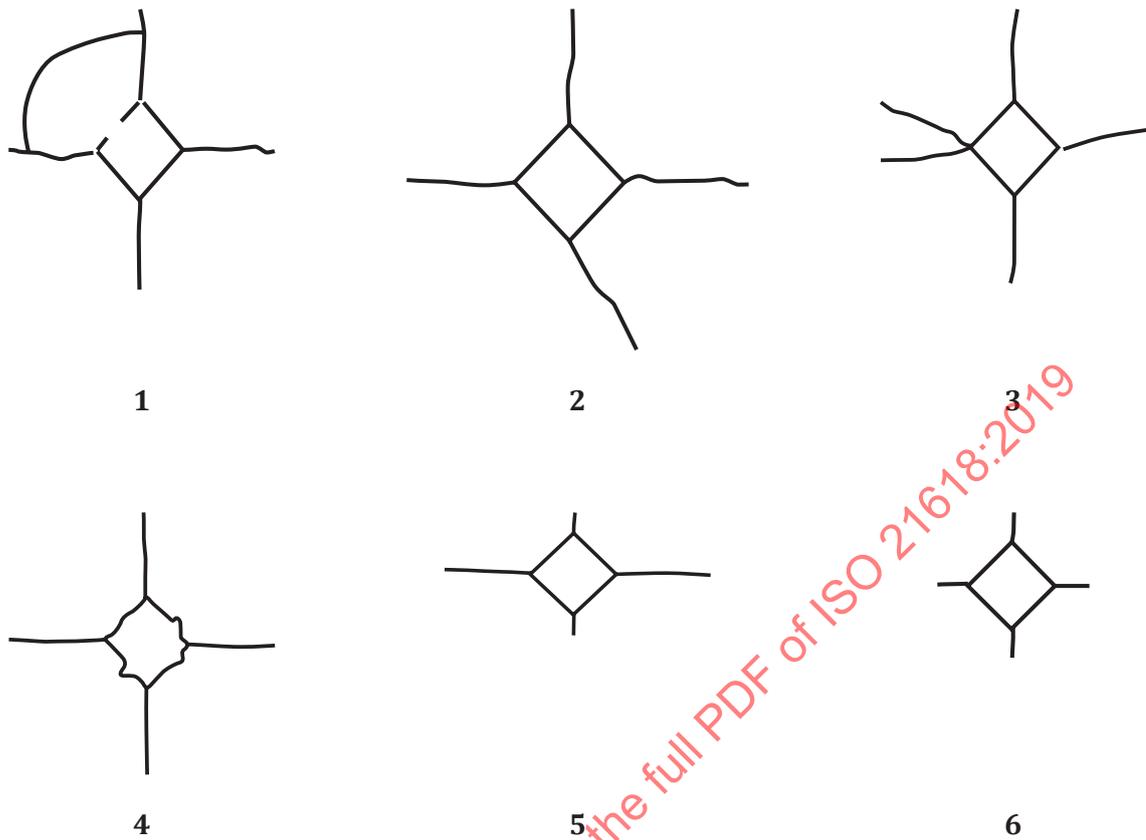
4 chipping and ragged edges
5 $2c_2 \ll 2c_1$ or $2c_1 \ll 2c_2$
6 $2c/2a < 2,5$

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.
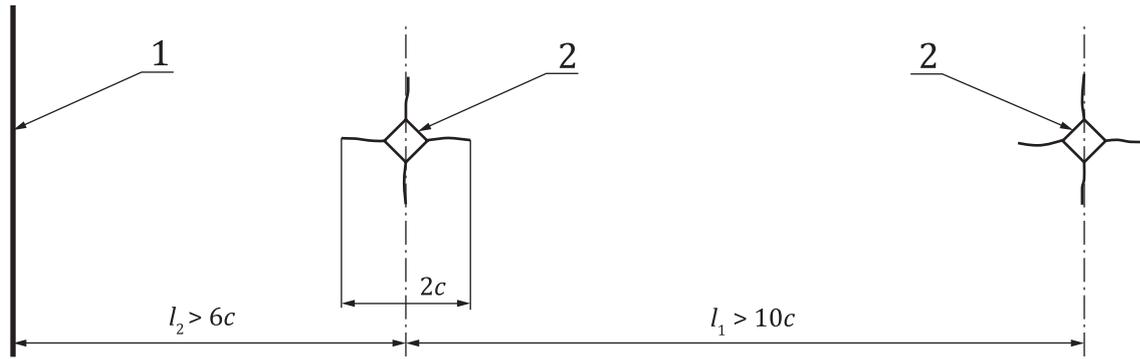
Key

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 described 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.

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.
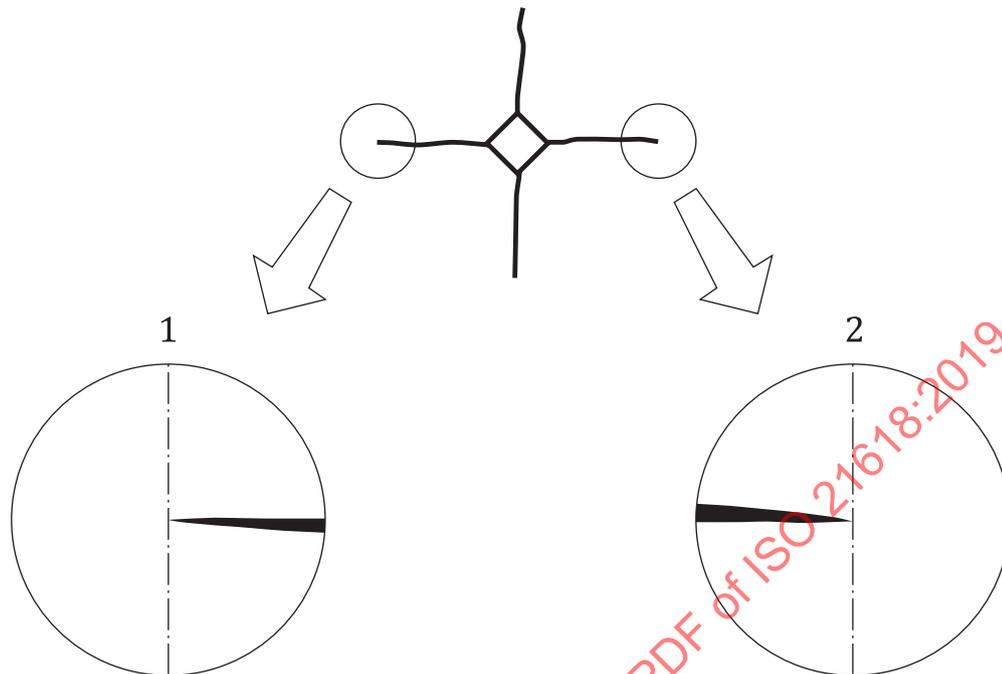
Key

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.
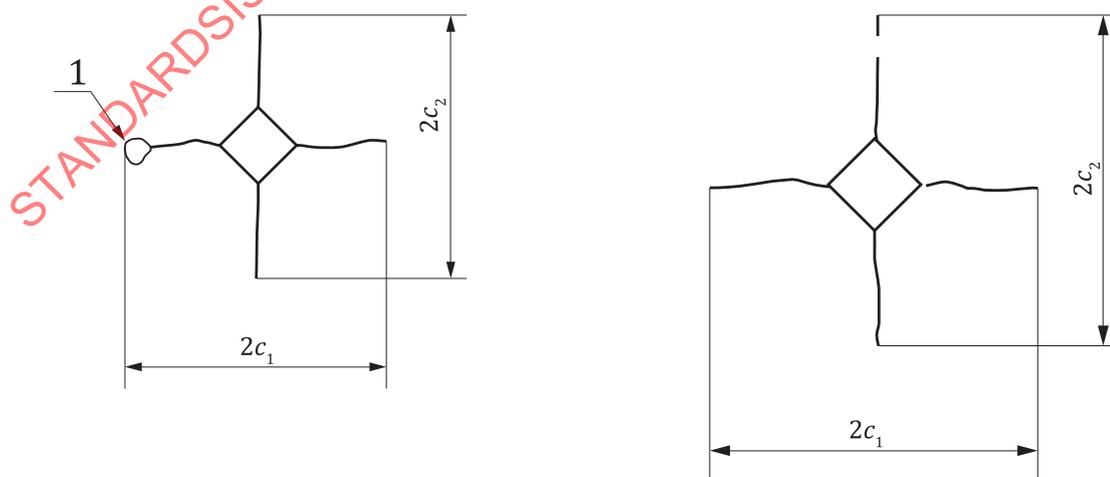
Key

1   void with a diameter of less than 10 μm

Figure 5 — Measurement method for the crack length when the crack tip touches a void or when the crack is disconnected at a grain or void and appears again behind the grain or void

8   Calculation

8.1   For each indentation, calculate the mean values of $2a$ and $2c$ according to Formula (1).

$$2a = \frac{2a_1 + 2a_2}{2} \quad \text{and} \quad 2c = \frac{2c_1 + 2c_2}{2} \tag{1}$$

8.2   For each indentation, calculate the Vickers hardness value according to Formula (2).

$$HV = 0{,}001\,854\frac{F}{(2a)^2} \tag{2}$$

where

HV is the Vickers hardness in gigapascals (GPa);

F is the applied force in newtons (N);

a is the mean half-length diagonal value in millimetres (mm).

8.3   For each indentation, calculate the indentation fracture resistance by Niihara's method according to Formula (3)[10].

$$K_{\mathrm{I,IFR}} = 0{,}000\,978\left(\frac{E}{HV}\right)^{0,4}\left(\frac{F}{c^{1,5}}\right) \tag{3}$$

where

$K_{\mathrm{I,IFR}}$ is the indentation fracture resistance in megapascals-square root meter (MPa·m$^{1/2}$);

E is the elastic modulus in gigapascals (GPa);

HV is the Vickers hardness in gigapascals (GPa);

F is the applied force in newtons (N);

c is the mean half crack length in millimetres (mm).

A sonic resonance method in accordance with ISO 17561 shall be used to measure the elastic modulus of the specimen provided that the test piece is available from the same material lot as the specimen. If the test piece for the sonic resonance method is not available from the same material lot, using literature data is permitted.

NOTE   The Niihara equation was selected because $K_{\mathrm{I,IFR}}$ calculated using this equation gives a relatively good correlation with K_{IC} measured using the SEPB or SCF methods for several structural ceramics, except for very fine-grained ceramics (see References [11],[12] and [13]).

9 Test report

The test report shall be prepared in accordance with ISO/IEC 17025, unless there are valid reasons for not doing so. The report of the results of an indentation fracture resistance test shall include the following items:

a) the name and address of the testing establishment;

b) the date of the test, a unique identification of the report and of each page, customer name and address and signatory of the report;

c) a reference to this document, i.e. determined in accordance with ISO 21618;

d) a description of the test material, elastic modulus, batch codes and date of manufacture, as appropriate;

e) geometry and dimensions of the test specimen;

f) sampling conditions of the test specimen from material and its machining conditions (when a specimen is heat treated, its conditions are included);

g) name and type of testing machine;

h) name and type of microscope;

i) magnification of objective lens and total magnification used to observe and measure the cracks;

j) environment of test, relative humidity and temperature;

k) indentation force;

l) elastic modulus used to calculate the indentation fracture resistance as well as the measuring method, i.e. determined with ISO 17561. If literature data were used, the source of the data should be provided;

m) a list of test results (number of valid indentations measured, as well as the total number of indentations, mean indentation fracture resistance and the standard deviation);

n) deviations from the specified procedures, if any.

The report of the results of an indentation fracture resistance test should preferably also include the following items:

o) kinds of additives and sintering method of test material;

p) mechanical properties of test material such as bending strength or Vickers hardness;

q) porosity of test material when slightly porous ceramics are employed.

Annex A
(informative)

General machining procedure for test piece preparation

A.1 General

The condition of the surface finish of the test piece affects the accuracy of the measurement of crack length. Detecting the correct position of the crack tip becomes difficult when small pits are made on the surface by dropout or fragmentation of a grain. Thus, it is necessary to adopt a proper machining process to avoid such difficulties. An example of a machining process for an ideal surface finish is described in this annex.

A.2 Cutting and grinding

A.2.1 Cut test pieces from a sintered bulk using a resin-bond diamond wheel, such as SDC140N-100B.

A.2.2 Prepare an iron-disc holder with a diameter of about 130 mm. Arrange multiple test pieces (more than five) along the circumference of the disc and glue the specimens with wax as shown in Figure A.1.
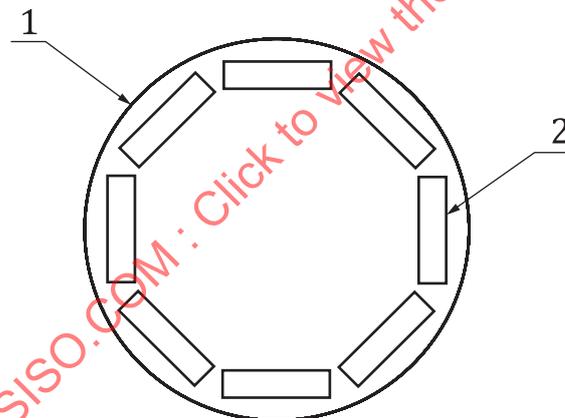
Key
1 iron-disc holder
2 test piece

Figure A.1 — Arrangement of test pieces on an iron-disc holder

A.2.3 Stick the iron holder to the table of the grinding machine with an electromagnet with the specimen side up.

A.2.4 A diamond-grid wheel 200 with a diameter of 200 mm shall be used to remove the last 0,2 mm or more on the surface. Set the speed of rotation of the wheel to 2 600 rpm. The depth of cut shall be less than 6 µm/pass. The number of spark out shall be 10.

A.2.5 Successively, a diamond-grid wheel 400 with a diameter of 200 mm shall be used to remove the last ca. 70 µm from the surface. Set the speed of rotation of the wheel to 2 600 rpm. The depth of cut shall be less than 3 µm/pass. The number of spark out shall be 10.

A.2.6   Remove the iron holder from the table of the grinding machine.

A.3   Polishing and lapping

A.3.1   Prepare a grooved polishing plate made of copper. The diameter of the plate should be 380 mm or more. Place the iron holder on the polishing plate with the sample side down. Set the speed of rotation of the plate to 60 rpm. The iron holder shall rotate on its axis. Spray a small amount of free abrasive polishing slurry with diamond grains of 3 μm onto the polishing plate once every minute. Adjust the pressure of the test piece to about 2 N/cm^2 by using an appropriate dead weight. The duration of the polishing shall be more than 1 h. The polishing is finished when all the grinding-induced flaws disappear.

A.3.2   Prepare a grooved lapping plate made of tin. The diameter of the plate should be 380 mm or more. Place the iron holder on the lapping plate with the sample side down. Set the speed of rotation of the plate to 60 rpm. The iron holder shall rotate on its axis. Spray a small amount of free abrasive lapping slurry with diamond grains of 0,5 μm onto the lapping plate once every minute. Adjust the pressure of the test piece to about 2 N/cm^2 by using an appropriate dead weight. The duration of the lapping shall be more than 1 h. Remove the last 10 μm or more from the surface during the polishing and lapping. The lapping is finished when a mirror-finish surface is obtained.

Annex B
(normative)

Adjustment of the axis of travelling-stage and calibration of the length measurement using the travelling stage

## B.1 General

Mismatch between the axis of the travelling stage and the field of optics causes an error in reading crack length. Thus, adjustment of the travelling stage is necessary before measurements. In addition, calibration of length measurement using the travelling stage is also required. The general procedures for both adjustment of the stage axis and calibration are described in this annex.

B.2 Adjustment of the axis of the travelling stage

B.2.1   Place a sample on the travelling stage of optics. Observe the sample with an objective lens of 40 × or 50 × and find a small and clear mark such as an impression or corner of the sample.

B.2.2   Set the mark on the horizontal line in the field of optics, for example a cross line of eyepiece [Figure B.1 a)].

a) Setting stage   b) Checking stage   c) Confirming stage
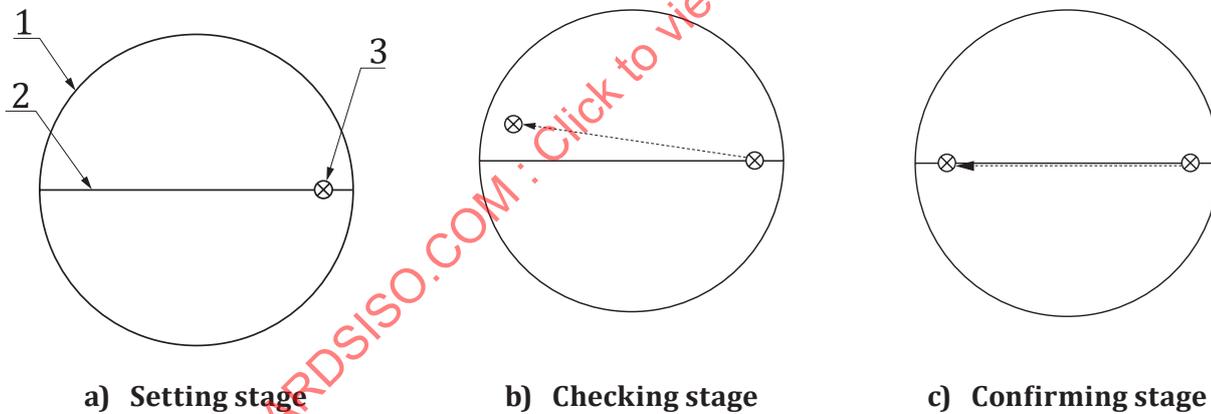
Key
1 field of optics
2 horizontal line
3 mark

Figure B.1 — Adjustment of the axis of travelling stage

B.2.3   Move the travelling stage so that the mark shifts to the left. If the mark deviates from the horizontal line [Figure B.1 b)], adjust the stage axis so that the mark shifts along the line [Figure B.1 c)].

B.3 Calibration of the length measurement using the travelling stage

B.3.1   Set an objective lens with a magnification of 40 × or 50 × to the optics.

B.3.2 Place an objective micrometer with length of 1 000 μm on the stage and observe it using the optical microscope.

B.3.3 Align the longitudinal direction of the micrometer to the horizontal direction in the field of optics.

B.3.4 Centre the left end of the micrometer and record the stage position.

B.3.5 Shift the stage horizontally until the right end of the micrometer is centred. Record the stage position.

B.3.6 Calculate the movement of the stage from the change of stage position. Check that the calculated movement of the stage is (1 000 ± 10) μm.

B.3.7 Calibrate the vertical length in the same manner as the horizontal procedure.