
**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**

*Céramiques techniques — Méthode d'essai de ténacité à la rupture des
céramiques monolithiques à température ambiante sur éprouvette
préfiissurée sur une seule face (méthode SEPB)*



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Contents

Page

Foreword	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	2
4 Symbols and designations	3
5 Principle	3
6 Apparatus	4
7 Test specimens	7
8 Test methods	8
9 Evaluation of validity of measured value	13
10 Calculation	14
11 Test report	15
Annex A (informative) Precracking fixture	16
Annex B (informative) Recommended procedures in SEPB method	18
Bibliography	21

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 15732 was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

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

1 Scope

This International Standard describes a test method for the determination of fracture toughness of monolithic ceramic materials at room temperature by the Single Edge Precracked Beam (SEPB) method.

This International Standard is intended for use with monolithic ceramics and whisker- or particulate-reinforced ceramics which are regarded as macroscopically homogeneous. It does not include continuous-fiber-reinforced ceramic composites.

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

Fracture toughness values determined with other test methods cannot be interchanged with K_{Ipb} as defined in this International Standard, and may not be interchangeable with each other.

Values expressed in this International Standard are in accordance with the International System of Units (SI).

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 1101:1983, *Technical drawings — Geometrical tolerancing — Tolerancing of form, orientation, location and run-out — Generalities, definitions, symbols, indications on drawings*

ISO 3312:1987, *Sintered metal materials and hardmetals — Determination of Young modulus*

ISO 4287:1997, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*

ISO 6507-1:1997, *Metallic materials — Vickers hardness test — Part 1: Test method*

3 Terms and definitions

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

3.1

stress intensity factor

K_I

magnitude of the elastic stress field singularity at the tip of a crack subjected to opening mode displacement

NOTE It is a function of applied force and test specimen size, geometry and crack length, and has the dimensions of force times length^{-3/2}.

3.2

fracture toughness

generic term for the magnitude of resistance to crack extension

3.3

fracture toughness value

K_{Ipb}

fracture toughness value measured by the SEPB method

NOTE This represents the measured stress intensity factor corresponding to the extension resistance of a straight-through pop-in crack formed via bridge loading of a Vickers indent or a saw notch. The measurement is performed in accordance with the operational procedure described in Clauses 5 and 10 and satisfies all the validity requirements.

3.4

precrack

crack induced artificially into a specimen, primarily so as to measure the fracture toughness

3.5

precrack front line

line to indicate the position of the tip of the precrack

3.6

pop-in

phenomenon where a crack arrests after sudden and unstable growth giving rise to an acoustic signature

3.7

three-point bending

loading configuration where a beam specimen is loaded at a location midway between two support pins

3.8

four-point bending

loading configuration where a beam specimen is symmetrically loaded at two locations that are situated one quarter of the overall span away from the outer two support pins

3.9

compliance

reciprocal of the gradient of the load versus deflection curve

NOTE Accordingly, as the crack extends, the increase of deflection results in an increase of compliance.

4 Symbols and designations

The symbols used throughout this International Standard and their designations are given in Table 1.

Table 1 — Symbols and designations

Symbol	Unit	Designation	References
b	mm	Width of central groove in anvil	A.2.3, Fig. A.1
d	mm	Thickness of specimen	7.1, Fig. 3
d_1	mm	Distance between supporting roller pins in bend test fixture (lower span)	6.4, Fig. 2
d_2	mm	Distance between loading roller pins in four-point bend test fixture (upper span)	6.4, Fig. 2
K_{Ipb}	MPa·m ^{1/2}	Critical stress intensity factor measured by the SEPB method	Clause 10, Eqs. 7 and 10
l	mm	Length of precrack	8.7.3, Fig. 6, Eq. 2
Δl	mm	Stable crack growth length	8.7.4, Fig. 6, Eq. 3
L	mm	Length of specimen	7.1, Fig. 3
L_a	mm	Length of bottom surface of specimen positioning groove of anvil (including the width, b , of central groove)	A.2.3, Fig. A.1
L_p	mm	Length of lower surface of loading plate	A.2.3, Fig. A.1
P_f	N	Maximum load during fracture of specimen	8.5.3, Fig. 5
w	mm	Width (depth) of specimen	7.1, Fig. 3
$\lambda\Delta l / \lambda l$	1	Compliance change	8.6, Eq. 1

5 Principle

This method is to obtain the fracture toughness value, K_{Ipb} , from the precrack length, 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 beam specimen. A straight-through pop-in precrack is induced in the specimen via bridge loading of a Vickers indent or a saw notch. Generally, this test is carried out under conditions of ambient temperature and environment.

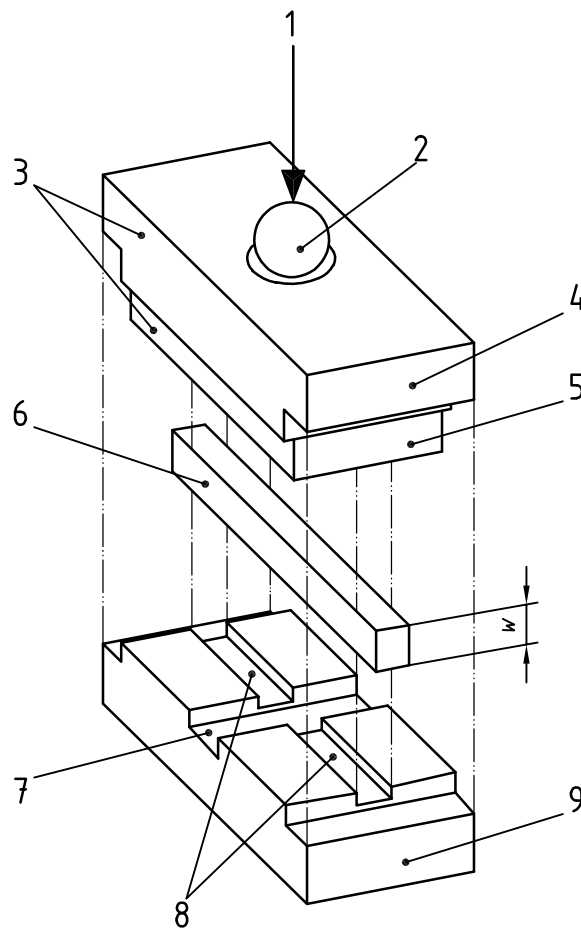
6 Apparatus

6.1 Precracking fixture

An appropriate apparatus is required to induce a pop-in precrack in the specimen in such a way that the crack front is approximately parallel to the specimen surface.

An example of the basic components of the bridge compression precracking fixture are a loading plate with a ball seat, an anvil with a central groove and a specimen-positioning groove, and a loading ball as shown in Figure 1. The shapes of the loading plate and the anvil to be used are symmetric from right to left, and from front to rear, and have depth exceeding at least three times the thickness of the specimen, d . The horizontal distance between the centre of the loading ball and the centre of the anvil is less than 0,1 mm.

NOTE Annex A contains recommendations for the typical design of a suitable bridge compression fixture that has been found to work satisfactorily for most types of ceramic materials.



Key

- | | | | |
|---|---|---|--|
| 1 | compression load | 5 | lower loading plate; e.g., of silicon nitride with mirror polished lower surface (joined to upper loading plate) |
| 2 | loading ball | 6 | specimen |
| 3 | loading plate | 7 | central groove |
| 4 | upper loading plate; e.g., of hardened steel with ball seat | 8 | specimen positioning groove |
| | | 9 | anvil; e.g., of hardened steel (HV 10 > 5 GPa) |

Figure 1 — Example of bridge compression precracking fixture

6.2 Precrack introduction loading apparatus

The loading apparatus shall be capable of smoothly applying the compression load to the fixture. High load accuracy is not required.

6.3 Bend testing machine

A material testing machine capable of maintaining the cross-head-speed constant shall be used. The accuracy of load measurement shall be $\pm 1\%$ over the entire range of load.

The rigidity of the entire testing system, including the bending test fixture specified in 6.4, shall be 3 MN/m or more against the load applied to the bending test fixture.

The rigidity of the entire testing system, including the testing machine, loading rods and bending test fixture, should be evaluated in accordance with Annex B.

6.4 Bend test fixture

The general features of the bend test fixtures are illustrated in Figure 2. The bend test fixture shall be symmetrical about the centre line shown and have a depth exceeding at least three times the thickness, d , of the specimen to be used. The fixture is designed to minimize frictional effects by allowing the support roller to roll apart slightly as the specimen is loaded, thus permitting rolling contact and avoiding frictional wedging of the specimen.

The roller pins are placed in the positioning grooves of the support member and of the loading member as shown in Figure 2. The rollers shall be parallel to each other to within 0,015 mm over a length equal to the specimen thickness, d . Other types of fixtures are acceptable, however, roller pins shall be free to roll. The length of each roller pin shall be equal to at least three times the specimen thickness. Materials composing the parts of the roller pins to be used shall have a modulus of elasticity not less than 196 GPa, as defined in ISO 3312 and a hardness of not less than 5 GPa Vickers (HV10) as defined in ISO 6507-1, and made of a material free from plastic deformation and risk of fracture. The radius of curvature of rollers and the distance between the rollers shall be as shown in Figure 2. The surface roughness of the rollers, R_a , as defined in ISO 4287 shall be not more than 0,4 μm .

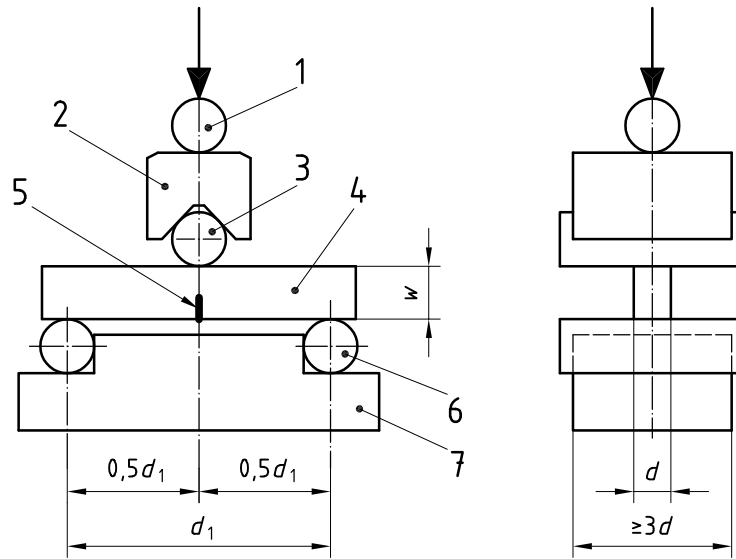
6.5 Compliance change measuring device

At the time of the bend test, the relation between the deflection of the test specimen and the load shall be measured by using the deflection meter which can measure the load point deflection between the centre of two supporting roller pins of the bend test fixtures and the centre of loading roller pin(s). The deflection measuring device shall have a resolution greater than 0,001 mm, and shall be calibrated to read within 0,001 mm of the true displacement. Measurements of displacement shall be made to a precision of 0,001 mm.

6.6 Measuring instruments

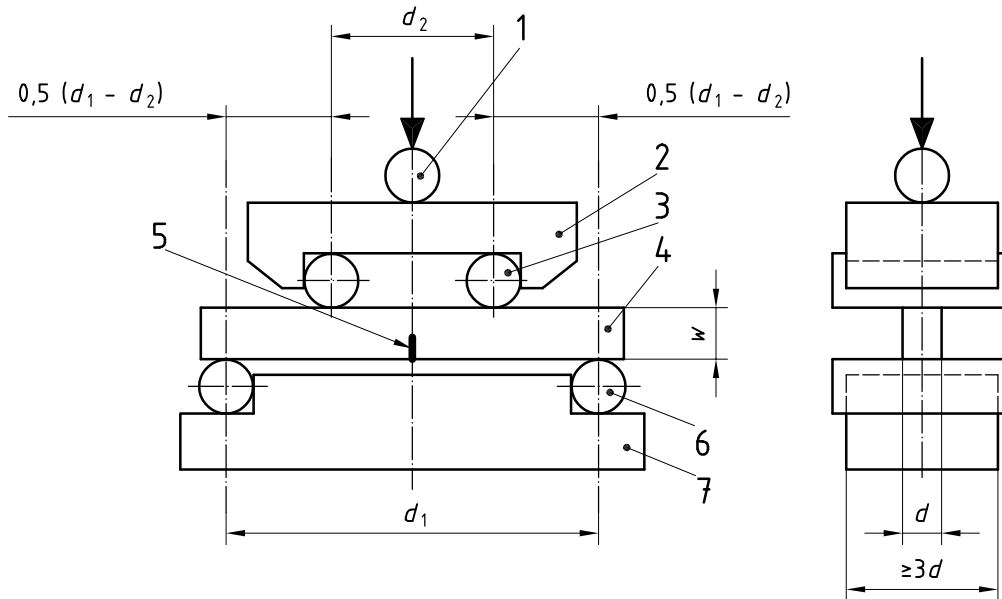
Three fundamental measurements are necessary for the calculation of K_{Ipb} , namely, the width, w , the thickness, d , and the precrack length, l .

Measuring devices such as micrometers or other devices having an accuracy of at least 0,01 mm shall be used for measuring the linear dimensions.



- Key**
- | | |
|----------------------|--------------------------|
| 1 loading ball | 4 specimen (I or II) |
| 2 loading member | 5 precrack |
| 3 loading roller pin | 6 supporting roller pins |
| | 7 support member |

a) Three-point bend test fixture



- Key**
- | | |
|-----------------------|--------------------------|
| 1 loading ball | 4 specimen (II or III) |
| 2 loading member | 5 precrack |
| 3 loading roller pins | 6 supporting roller pins |
| | 7 support member |

b) Four-point bend test fixture

Dimensions in millimetres

Bending mode	Specimen	Diameters of roller pins	d_1 (lower span)	d_2 (upper span)
3-point bend	I	4,0 to 5,0	$16 \pm 0,2$	–
3-point bend	II	4,0 to 5,0	$30 \pm 0,3$	–
4-point bend	II	4,0 to 5,0	$30 \pm 0,3$	$10 \pm 0,2$
4-point bend	III	4,0 to 5,0	$40 \pm 0,4$	$20 \pm 0,2$

Figure 2 — Bend test fixtures

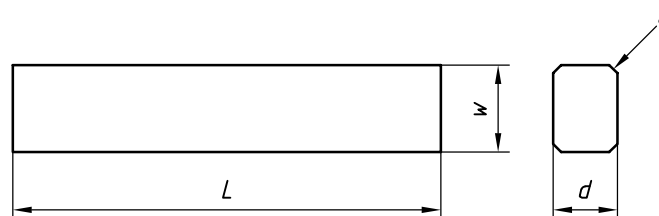
7 Test specimens

7.1 Shape and dimensions of specimen, and chamfering of edge

The shape of the specimens shall be that of a rectangular beam and its dimensions shall be as shown in Figure 3. Sampling position and orientation of specimen removal from raw material shall be recorded.

The opposing faces of the specimen shall be parallel to each other and the faces shall intersect perpendicularly. The maximum variation in parallelism and perpendicularity shall not exceed 0,01 mm as defined in ISO 1101.

Further, the four long edges of each specimen shall be chamfered uniformly at $45^\circ \pm 5^\circ$. The chamfered edge length shall be $0,12 \text{ mm} \pm 0,03 \text{ mm}$, as shown in Figure 3 (hereafter, a specimen of 18 mm or more in length is referred to as “specimen I”, a specimen of 36 mm or more in length, as “specimen II”, and a specimen of 45 mm or more in length, as “specimen III”).



Dimensions in millimetres

Specimen	Total length, L	Width, w	Thickness, d	Chamfering, C
I	≥ 18	$4 \pm 0,1$	$3 \pm 0,1$	$0,12 \pm 0,03$
II	≥ 36	$4 \pm 0,1$	$3 \pm 0,1$	$0,12 \pm 0,03$
III	≥ 45	$4 \pm 0,1$	$3 \pm 0,1$	$0,12 \pm 0,03$

Figure 3 — Dimensions of specimen

7.2 Surface roughness of upper and lower surfaces and both side surfaces of specimen

The surface roughness of the four surfaces of the specimen, R_a , as defined in ISO 4287 excluding both end surfaces in the length direction shall be not more than 0,2 μm .

7.3 Number of specimens

The number of specimens shall be not less than five pieces. All specimens shall comply with the specification given in 9.1.

8 Test methods

8.1 Measurement of thickness and width of specimen

The measurement of thickness and width of specimen shall be carried out using a micrometer or other device accurate to the nearest 0,01 mm.

8.2 Introduction of precrack starter

8.2.1 General

As the precrack generating start point, a Vickers indentation (or Knoop indentation) or notch near the middle of the $d \times L$ surface of the specimen in a direction within 2° of the normal direction to the $w \times L$ surface shall be induced according to the schematic diagram in Figure 4. The recommended conditions for introduction of the precrack starter are given in 8.2.2 and 8.2.3.

NOTE The position of the precrack starter in the lengthwise direction is not specified, but the positioning is important for precrack introduction (see Annex B).

8.2.2 The use of Vickers indentation (or Knoop indentation)

8.2.2.1 Position of indentation

One point shall be at the middle of the $d \times L$ plane of the specimen. Additional points shall be placed symmetrically on both sides of the first indent.

8.2.2.2 Direction of indentation

The diagonal line of the Vickers indentation (or major axis of Knoop indentation) shall intersect perpendicularly the lengthwise direction of specimen.

8.2.2.3 Indentation load

Select an indentation force adequate to produce straight cracks from the corners of the indentation without causing extensive ancillary damage.

An indentation force of 98 N (or 100 N) is adequate to induce a starter crack in most materials. If this force produces excessive damage, e.g. in relative soft or brittle materials, use a lower force. If precracking using an indentation prepared using a force of 98 N (or 100 N) is unreliable, increase the number of adjoining indentations across the specimen width rather than increasing the force. However, where introduction of a precrack is impossible, the notch method may be used.

8.2.3 The use of a straight-through saw notch

8.2.3.1 Width of notch

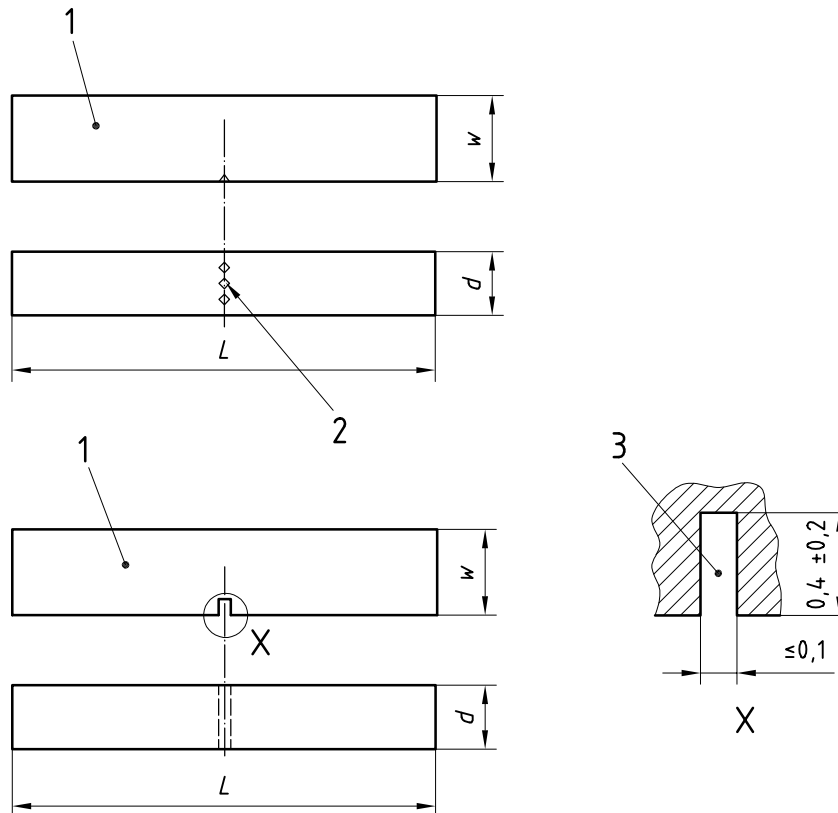
The width of the notch shall be not more than 0,1 mm. The shape of the tip of the notch is not specified.

8.2.3.2 Depth of notch

The depth of the notch shall not exceed 0,6 mm and be $0,4 \text{ mm} \pm 0,2 \text{ mm}$.

NOTE If a notch depth exceeding 0,6 mm is used, stable crack growth, instead of pop-in, may occur from the notch tip during precrack introduction. Fracture toughness values measured from the stably grown precrack might be greater than the fracture toughness measured under this procedure.

Dimensions in millimetres



Key

- 1 specimen
- 2 Vickers (or Knoop) indentation(s)
- 3 notch

Figure 4 — Introduction of Vickers indentation or notch

8.3 Introduction of precrack

8.3.1 Arrange the specimen with the previously induced precrack starter, between the loading plate of the precrack inducing fixture and the anvil as shown in Figure 1. Introduce an unstably grown pop-in crack. The $d \times L$ surface contacts with the loading plate and the specimen positioning groove of the anvil. The procedure is given in 8.3.2 to 8.3.5.

NOTE A recommended precracking fixture is shown in detail in the Annex A. A recommended procedure for precrack introduction is shown in detail in the Annex B.

8.3.2 Sufficiently clean the specimen, the bottom surface of specimen positioning groove of the precracking anvil and the lower surface of loading plate by wiping off any oil and stains using acetone. Place the specimen in the specimen positioning groove of the anvil as shown in Figure 1, taking care to arrange the specimen so that the lengthwise direction of the specimen intersects perpendicularly the central groove of the anvil, and the precrack starter positions in the range of $\pm 0,1$ mm from the centre of the central groove.

8.3.3 Place the loading plate on the specimen, making sure that the specimen does not slide. The loading plate and the anvil shall be arranged symmetrically.

8.3.4 Using the loading ball and anvil, increase the compression load vertically to the loading plate. Stop loading immediately after the pop-in sound is detected, because an excessive compression load may damage the fixtures and cause uneven development of precrack. The speed at which to apply a compression load is recommended to be in the range 300 N/s to 1 000 N/s.

NOTE 1 For materials with a rising R-curve the measured fracture toughness value might be artificially high if stable crack growth occurs after pop-in.

NOTE 2 Sonic sensors, e.g. an acoustic emission sensor or a stethoscope, are useful for detecting the pop-in during precracking. See Annex B.

8.3.5 Remove the specimen from the fixture and confirm that the precrack has been induced on both side surfaces. If the precrack is difficult to locate, a dye penetrant, e.g. oil based paint mixed with acetone, may be used to improve visibility. If a dye penetrant is used, the following bend test shall be carried out after complete drying of the dye penetrant.

8.4 Test atmosphere

The bend test is carried out under conditions of ambient temperature and environment. If susceptibility to environmental degradation, such as slow crack growth during the bend test, is a concern, environmental effects shall be evaluated by measuring both the compliance change and the stable crack growth length specified in 8.6 and 8.7. The permissible range of the compliance change and the stable crack growth length in order to evaluate the valid fracture toughness value, are specified in 9.2.

In many ceramics, including most oxides, moisture in the atmosphere can have a marked effect in enhancing crack propagation characteristics, and hence in reducing the measured toughness. Environment conditions, such as relative humidity and temperature, shall be reported. A dry atmosphere, such as dry air, nitrogen or argon with a purity of 99,9 % or better at atmospheric pressure, or vacuum of less than 0,13 Pa, removes most of the effects of moisture.

8.5 Three- or four-point bending fracture test

8.5.1 General

Arrange the precracked specimen within the bend test device as shown in Figure 2. Ensure that the $d \times L$ surface of the specimen makes contact with the supporting roller pins, and that the roller pins are in the correct starting position. The procedures are described in 8.5.2 and 8.5.3.

8.5.2 Arrangement of specimen

Ensure the specimen is dry. Arrange the specimen so that the roller pins lie with their axes within 2° perpendicular to the length of the specimen. The distance between the position of precrack starter and the dead centre of the loading pin(s) shown in Figure 2 shall be within 1 % of the lower span length, d_1 .

8.5.3 Bending fracture test

Apply a compressive load to the loading pin(s) at a cross-head speed of 0,5 mm/min until the specimen fractures, and measure the maximum load, P_f , to an accuracy of $\pm 1\%$ and the compliance change as specified in 8.6. The time to failure should not exceed 20 s in order to avoid environmental effects.

8.6 Measurement of compliance change

Generally, a compliance change in a load versus load point deflection curve during a flexural test is not observed for common monolithic ceramics. However, if observed, the validity of measured fracture toughness values shall be evaluated. In this international Standard, the validity is evaluated from the movement of the pop-in precrack front line before final fracture.

Measure the load point deflection between the centre of two supporting roller pins of the bend test fixture and the loading pin(s), using a suitable deflection meter. Follow the procedure shown in Figure 5 to measure the compliance change $\lambda\Delta l / \lambda l$. The minimum reading in this case shall be 0,001 mm, or the accuracy shall be equivalent to or better than this. When the deflection is measured, it shall not adversely affect the measurement of fracture load of test specimen. Use the following equation to obtain $\lambda\Delta l / \lambda l$. That value shall be the compliance change.

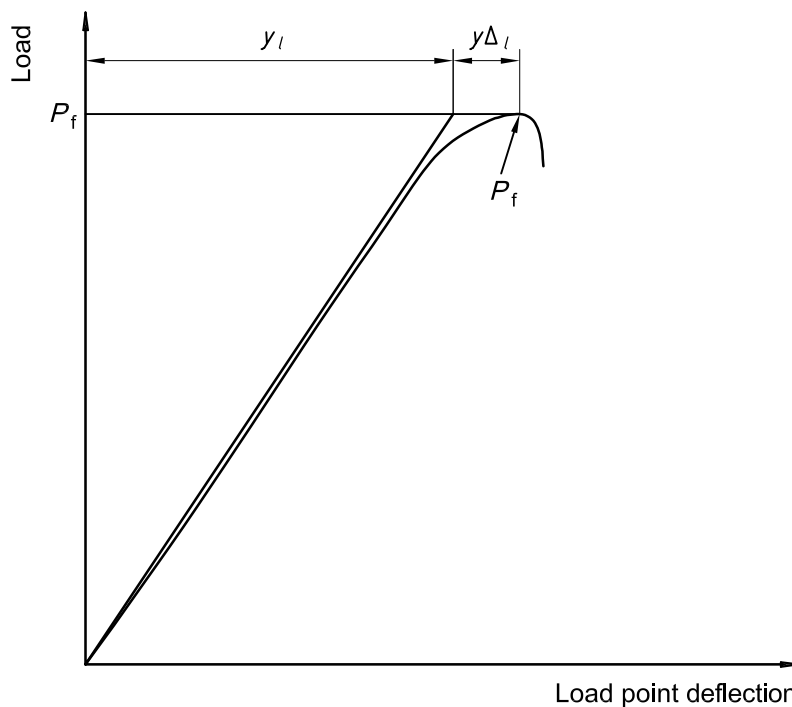
$$\frac{\lambda\Delta l}{\lambda l} = \frac{y\Delta l / P_f}{y l / P_f} = \frac{y\Delta l}{y l} \quad (1)$$

where

P_f is the maximum load before fracture in newtons;

$y l$ is the deflection where the extension line of load line reaches P_f , in millimetres;

$y\Delta l$ is a value obtained by subtracting $y l$ from deflection at the time of fracture, in millimetres.



$$\frac{\lambda\Delta l}{\lambda l} = \frac{y\Delta l / P_f}{y l / P_f} = \frac{y\Delta l}{y l} \leq 0,1 l/w$$

Figure 5 — Load versus load point deflection curve and compliance change at bend test

8.7 Measurement of precrack length and stable crack growth length

8.7.1 Enlarge the fracture surface of the fractured specimen by at least $\times 20$ using a microscope or by photography, and measure the length of precrack to the nearest 0,01 mm. If stable crack growth of a different pattern is observed between the precracked region and the unstable crack growth region on the fracture surface, measure the stable crack growth length in the same way. The measurement of precrack length and stable crack growth length, and procedures of calculation, are given in 8.7.2 to 8.7.4.

A calibration of micrograph system shall be prepared from the measurement of the known specimen width in order to determine the true magnification used for the fracture surface.

NOTE The stable crack growth region is generally observed on the fracture surface by having different light reflection or a different colour (generally whitish).

8.7.2 As shown in Figure 6, the distance from the lower surface of the specimen to the front line of the precrack in the specimen width, w , direction is determined by measuring the lengths of $l_1, l_2, l_3, \Delta l_1, \Delta l_2$ and Δl_3 on the three lines which divide into four equal parts the thickness, d , of the specimen. In the case where the front line of the precrack cannot be judged from the fracture surface profile, consider the front line of the dyed region to be the front line of precrack.

8.7.3 Obtain the average value of each measured value according to the following formula and take it as the precrack length.

$$l = \frac{l_1 + l_2 + l_3}{3} \tag{2}$$

where l_1, l_2 and l_3 are measured pop-in crack lengths in millimetres.

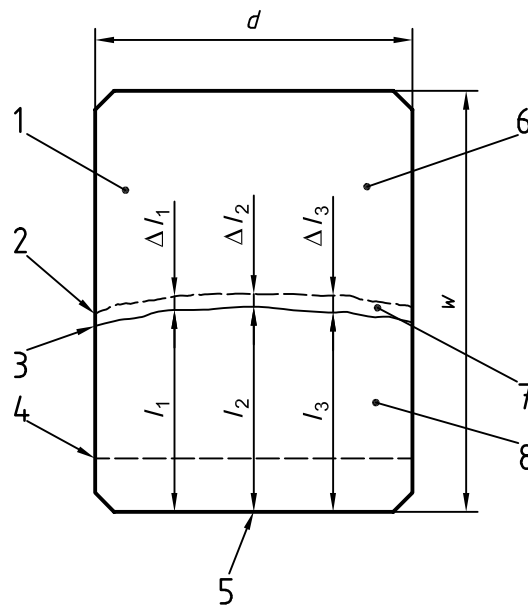
Thus l is the average precrack length in millimetres.

8.7.4 Obtain the average value of each measured value, $\Delta l_1, \Delta l_2$ and Δl_3 in accordance with the following formula and take it as the stable crack growth length.

$$\Delta l = \frac{\Delta l_1 + \Delta l_2 + \Delta l_3}{3} \tag{3}$$

where $\Delta l_1, \Delta l_2$ and Δl_3 are measured stable crack growth lengths in millimetres.

Thus Δl is average stable crack growth length in millimetres.



Key

- | | |
|----------------------------------|--------------------------------|
| 1 fracture surface of specimen | 5 Vickers impression(s) |
| 2 stable growth crack front line | 6 unstable crack growth region |
| 3 precrack front line | 7 stable crack growth region |
| 4 notch | 8 pop-in crack growth region |

Figure 6 — Measurement of length of precrack and stable crack growth

9 Evaluation of validity of measured value

9.1 Specifications of precrack dimensions and shape

9.1.1 General

The tolerance ranges of inclination, oblique advance, and length of front line of the precrack are specified as follows. Results are acceptable only for specimens which comply with all of the specifications given in 9.1.2 to 9.1.4. The result obtained from unacceptable test specimen shall be recorded only for informative reference value.

9.1.2 Permissible range of inclination of precrack front line

The difference between maximum and minimum measured values of l shall be no greater than 10 % of the average precrack length, l . See Equation (4).

$$\frac{l_{\max} - l_{\min}}{l} \leq 0,1 \quad (4)$$

where

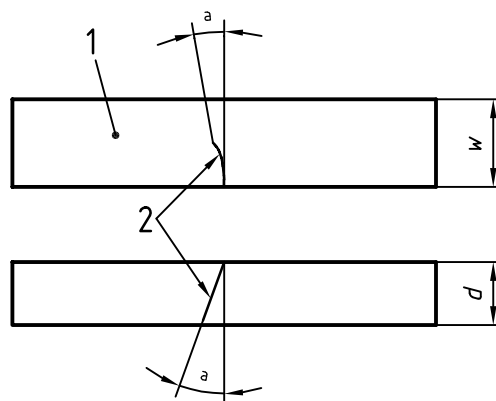
l_{\max} is the maximum measured value in l_1 , l_2 and l_3 ;

l_{\min} is the minimum measured value in l_1 , l_2 and l_3 .

9.1.3 Permissible range of precrack oblique advance

As shown in Figure 7, the advancing route of the precrack at the lower surface and both side surfaces of the specimen shall be not more than 5° from the surface normal to the thickness direction and width direction of the specimen for the 3-point bend specimen and not more than 10° for the 4-point bend specimen.

NOTE Acceptance or rejection of the result for a test piece, resulting from an angle crack may be determined most simply by using a microscope with a rotary stage, viewing the side face of the test piece, aligning a cross-wire first with the test piece edge, and then with the average line of the crack from its tip, in both cases reading the angular scale on the microscope stage to the nearest $0,5^\circ$. From the angle difference the crack misalignment angle may be computed taking into account twist.



Key

1 specimen

2 precrack

a 5° max. for the 3-point bend specimen; 10° max. for the 4-point bend specimen.

Figure 7 — Permissible range of oblique advance of precrack

9.1.4 Permissible range of precrack length

The precrack length shall be in the range of $0,35 \leq l/w \leq 0,6$.

9.2 Provision for stable crack growth

9.2.1 General

The permissible range of the stable crack growth length and the compliance change shall be specified in 9.2.2 and 9.2.3 respectively. In principle, a test specimen which has passed all these provisions and the provision laid down in 9.1 shall be deemed as giving an acceptable result. The results obtained from unacceptable test specimens shall be recorded only for informative reference value.

9.2.2 Permissible range of stable crack growth length

The stable crack growth length, Δl , shall be within the range of 2 % of the precrack length, l .

$$\Delta l \leq 0,02l \quad (5)$$

9.2.3 Permissible range of compliance change

The compliance change, $\lambda \Delta l / \lambda l$, shall be within the range of 10 % of the ratio of precrack length, l , to the depth of test specimen, w .

$$\frac{\lambda \Delta l}{\lambda l} \leq 0,1 \frac{l}{w} \quad (6)$$

10 Calculation

Calculate the fracture toughness value, K_{Ipb} (critical stress intensity factor in mode I), in units of megapascal metres to the power half from the measured values for each specimen according to the following formulae. Obtain the average value of fracture toughness of five or more specimens complying with the specification given in 9.1 and 9.2 and round off to one decimal place.

$$K_{Ipb} = \frac{P_f \times d_1}{d \times w^{3/2}} \left[\frac{3}{2} \left(\frac{l}{w} \right)^{1/2} \times Y \left(\frac{l}{w} \right) \right] \quad (7)$$

where, in the case of 3-point bend specimen I (applicable range of formula: $0,35 \leq l/w \leq 0,6$),

$$Y \left(\frac{l}{w} \right) = \frac{1,99 - \frac{l}{w} \left(1 - \frac{l}{w} \right) \left[2,15 - 3,93 \frac{l}{w} + 2,7 \left(\frac{l}{w} \right)^2 \right]}{\left(1 + 2 \frac{l}{w} \right) \left(1 - \frac{l}{w} \right)^{3/2}} \quad (8)$$

In the case of 3-point bend specimen II (applicable range of formula: $0,35 \leq l/w \leq 0,6$),

$$Y \left(\frac{l}{w} \right) = 1,964 - 2,837 \frac{l}{w} + 13,711 \left(\frac{l}{w} \right)^2 - 23,250 \left(\frac{l}{w} \right)^3 + 24,129 \left(\frac{l}{w} \right)^4 \quad (9)$$

In the case of 4-point bend specimens II and III (applicable range of formula: $0,35 \leq l/w \leq 0,6$),

$$K_{Ipb} = \frac{P_f (d_1 - d_2)}{d \times w^{3/2}} \times \left[\frac{3 \left(\frac{l}{w} \right)^{1/2}}{2 \left(1 - \frac{l}{w} \right)^{3/2}} F \left(\frac{l}{w} \right) \right] \quad (10)$$

where

$$F \left(\frac{l}{w} \right) = 1,9887 - 1,326 \frac{l}{w} - \frac{\left[3,49 - 0,68 \frac{l}{w} + 1,35 \left(\frac{l}{w} \right)^2 \right] \left(\frac{l}{w} \right) \left(1 - \frac{l}{w} \right)}{\left(1 + \frac{l}{w} \right)^2} \quad (11)$$

11 Test report

The test report shall include the following information:

- a) type of material, code, batch number, date of manufacture;
- b) type of specimen (either specimen I, specimen II or specimen III);
- c) type of bending mode (either 3-point bend or 4-point bend);
- d) number of specimens complying with the specifications laid down in 9.1 and 9.2;
- e) measured fracture toughness values (critical stress intensity factor in mode I), K_{Ipb} , and the average, in megapascal metres to the power half;
- f) manufacturing process and dimensions, form of raw material as well as sampling position and direction of specimen;
- g) specimen dimensions: thickness, d , width, w , in millimetres;
- h) precracking conditions (type of precrack starter, width of anvil central groove and pop-in load, etc.);
- i) environment conditions, relative humidity and temperature;
- j) maximum load before fracture, P_f , in newtons;
- k) time to failure, in seconds;
- l) precrack lengths and their average, l , in millimetres;
- m) presence or absence of stable crack growth and average of stable crack growth length;
- n) presence or absence of compliance change and the value of compliance change.

Annex A (informative)

Precracking fixture

A.1 Introduction

While there are a number of methods for inducing a straight precrack in a ceramic specimen, the bridge compression method has been found to be reliable for a number of materials. This Annex provides for guidance details of typical compression fixture designs, which have been found to operate satisfactorily. It should be noted that whatever the details of the design, the result will depend to some extent on the skill of the user, and on the precision with which the fixture is made.

NOTE The recommended techniques relating to the precrack introduction fixture are noted in Annex B.

A.2 Recommended design

A.2.1 The basic components of the precracking fixture are a loading plate with a ball seat, an anvil with central groove and a loading ball as shown in Figure A.1. The shapes of the loading plate and the anvil to be used are symmetrical from right to left, and from front to rear, and have a depth exceeding at least three times the thickness of the specimen, d . The horizontal distance between the centre of the loading ball and the centre of the anvil is less than 0,1 mm. The width, b , of the central groove of the anvil should be between 2 mm and 8 mm and should be selected by trial and error so that the precrack length to be induced complies with the specification laid down in 9.1.

A.2.2 The anvil has a specimen-positioning groove that is at a right angle to the central groove. The bottom width of the specimen-positioning groove is larger than the thickness of the specimen, as shown in Figure A.1. The lower surface of the specimen should closely contact the bottom of the specimen-positioning groove.

A.2.3 In order to avoid stress concentration on the specimen ends, the length, L_p , of the lower surface of the loading plate and the length, L_a , of the bottom surface of the specimen-positioning groove of the anvil (including the width, b , of the central groove) should be shorter than the length of the specimen, L , and are made identical and equal to 18 mm ($\pm 0,1$ mm) which is shortest length in a type III specimen.

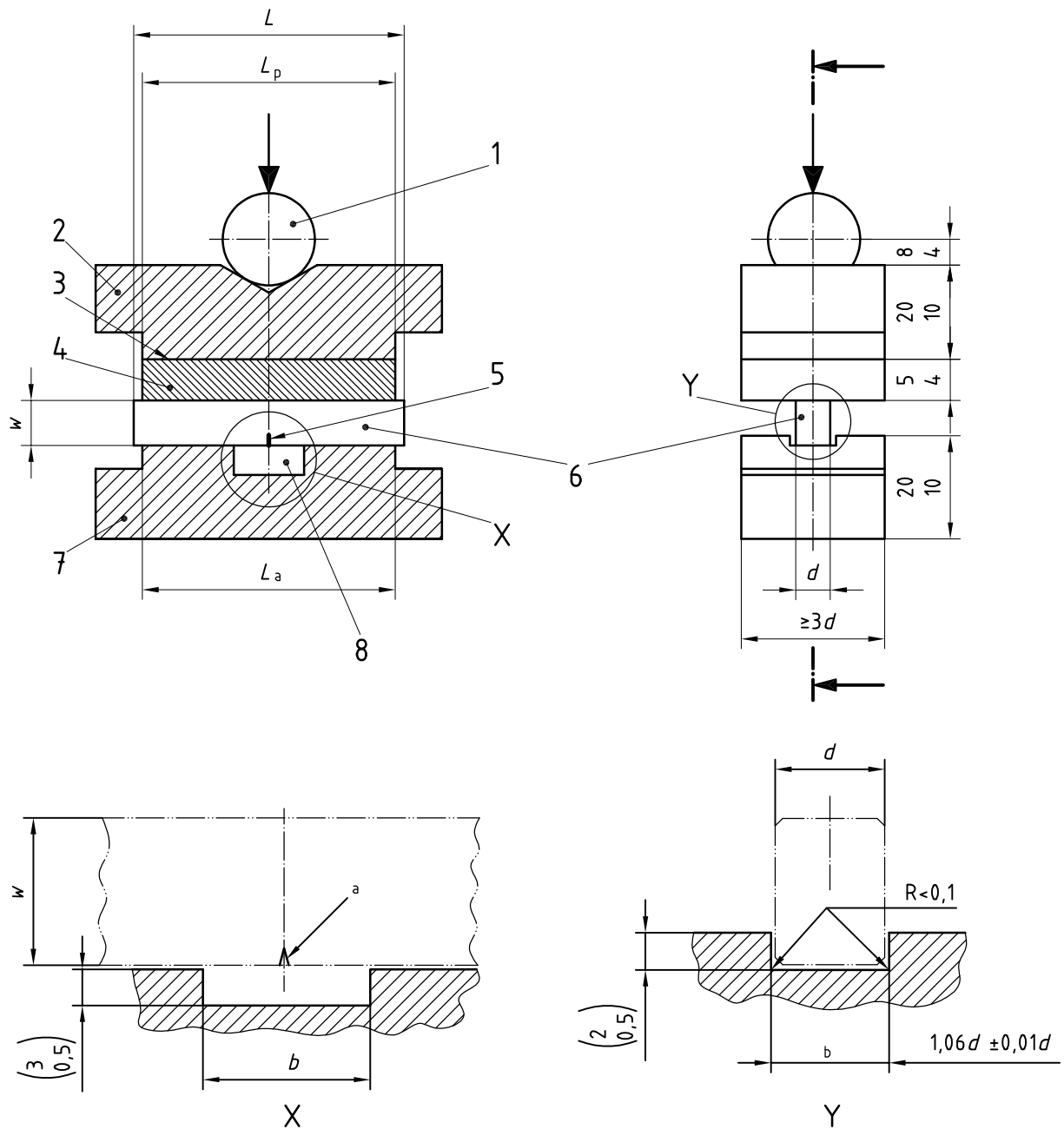
NOTE By using a precracking fixture with a fixed length $L_p (= L_a; \leq L)$ the pop-in load in each material is independent of the specimen types (I, II, III).

A.2.4 Materials composing the parts of the loading ball, the lower surface of the loading plate and bottom surface of the specimen positioning groove of the anvil in contact with the specimen should have a modulus of elasticity not less than 196 GPa as defined in ISO 3312 and a hardness of not less than 5 GPa in Vickers (HV10) as defined in ISO 6507-1, and be free from plastic deformation when the specimen is compressed.

A.2.5 The variation in flatness of the lower surface of the loading plate and of the bottom surface of the specimen-positioning groove of the anvil should preferably be not more than 0,01 mm as defined in ISO 1101, and their roughness should preferably be not more than 0,2 μm R_a as defined in ISO 4287. A sintered silicon nitride plate with a mirror finish and a thickness exceeding 4 mm is recommended for the bottom part of the loading plate. The upper and lower surfaces of the loading plate, the bottom surface of the specimen positioning groove and the lower surface of the anvil are parallel to one another. The deviation from parallelism as defined in ISO 1101 should not exceed 0,02 mm.

A.2.6 As an alternative, the surface of the loading plate in contact with the specimen may be rounded with a radius of curvature exceeding $3d$ and an axis lying along the length of the specimen. This arrangement has been found to improve the reliability of precracking by more closely defining the axis of loading. See [1],[2], [3].

Dimensions in millimetres



Key

- | | |
|--|--------------------------|
| 1 loading ball (diameter, 4 mm to 8 mm) | 5 precrack starter |
| 2 upper loading plate (hardened steel) | 6 specimen |
| 3 joint | 7 anvil (hardened steel) |
| 4 lower loading plate with mirror polished lower surface (silicon nitride) | 8 central groove |

- w* width of specimen ($4 \text{ mm} \pm 0,1 \text{ mm}$)
d thickness of specimen ($3 \text{ mm} \pm 0,1 \text{ mm}$)
L overall length of specimen ($L \geq L_p$)
 $L_p = L_a$ ($= 18 \text{ mm} \pm 0,1 \text{ mm}$)
b width of central groove (2 mm to 8 mm)

- a* Precrack starter positions in the range of $\pm 0,1$ mm from centre of central groove.
b Width of bottom flat surface shall exceed *d*.

Figure A.1 — Precrack inducing fixture

Annex B (informative)

Recommended procedures in SEPB method

B.1 Precrack introduction fixture

B.1.1 The pop-in crack length is dependent upon the fracture toughness of the material, the type of precrack starter, the introduction conditions and the width of the central groove of the anvil. Therefore, it is necessary to select the width of the central groove of the anvil by trial and error corresponding to the test material so as for it to be in the range specified in 9.1. However, an anvil with variable groove width exhibits insufficient rigidity, and therefore several anvils with different groove widths should preferably be prepared (the range of groove width is usually 2 mm to 8 mm). Some examples are shown in Table B.1.

Table B.1 — Some examples of precracking conditions and obtained pop-in crack length

Materials	Groove width of anvil	Pop-in load	Pop-in crack length
Alumina ($K_{Ipb} = 4 \text{ MPa}\cdot\text{m}^{1/2}$)	4 mm	8 kN	1,5 mm
	5 mm	7 kN	1,8 mm
Silicon nitride ($K_{Ipb} = 5 \text{ MPa}\cdot\text{m}^{1/2}$)	4 mm	12 kN	1,7 mm
	5 mm	10 kN	2,0 mm
P.S.Z. ($K_{Ipb} = 6 \text{ MPa}\cdot\text{m}^{1/2}$)	5 mm	25 kN	1,4 mm
	6 mm	20 kN	1,8 mm
Silicon carbide ($K_{Ipb} = 3 \text{ MPa}\cdot\text{m}^{1/2}$)	3 mm	3 kN	2,1 mm
	4 mm	2 kN	2,4 mm

NOTE The data are independent of the specimen type (I, II, III). The data were obtained using the specimen with one Vickers indentation as precrack starter and using the precracking fixture with mirror-polished Si_3N_4 loading plate and hardened steel anvil ($L_p = L_a = 18 \text{ mm}$).

B.1.2 The loading plate is not required to be monolithic, and when a plate of high-performance ceramic, e.g. sintered silicon nitride, is adhered to the part in contact with the specimens, the fixture may be used for a long time. In order to ensure that the mutual positioning of loading plate and the anvil are correct, auxiliary parts such as an outer frame are useful. In order to arrange correctly the precrack starter at the centre of the central groove of the anvil, it is convenient to prepare such parts.

B.2 Introducing positions of precrack starter and precrack

The important points in Figures 1, 2 and A.1 are the positions of the precrack starter and the precrack; it is not required that the specimen be placed symmetrically on the anvil, and further, not required that the anvil be exchanged when using different specimen lengths. However, in order to avoid unintentional stress concentration, the specimen must be longer than, or equal to, the length of the specimen positioning groove of the anvil and loading plate. The condition is that the precrack starter should be in the centre of the anvil groove.

B.3 Width and depth of notch

The width of the notch is generally specified to be not more than 0,1 mm, but if the width is made narrower, the pop-in is effected more easily. For a notch depth of 0,6 mm or more pop-in may not occur and the crack may develop as stable crack growth and therefore it is not recommended.

B.4 Introduction of precrack

When the introduction of the precrack is performed in a noisy environment, if a sonic sensor, e.g., an acoustic emission sensor or a stethoscope, is connected to the precracking fixture, detection of pop-in signal is made easy. Because stress corrosion cracking due to water vapour in atmospheric air may occur, the rate of increase of load should preferably be not less than 294 N/s. In the case of a high toughness material in which the pop-in crack is difficult to induce, aluminium foil may be placed between the upper surface of the specimen and the lower surface of the loading plate to be compressed.

B.5 Confirmation of precrack introduction and measurement of precrack length

B.5.1 In the case of a material, such as alumina, zirconia, etc., in which it is difficult to see the precrack, it is preferable to introduce a dye penetrant such as oil paint dissolved in acetone into the precrack.

B.5.2 Further, in the case where the length of introduced precrack exceeds the specified upper limit, it is doubtful that stable crack growth will occur during the fracture toughness test. For materials with a rising R-curve the measured fracture toughness value might be artificially high if the stable crack growth occurs after pop-in.

B.6 Evaluation of rigidity of testing system

The rigidity of the entire testing system, including the testing machine, loading rods and bend test fixture, shall be evaluated using a rectangular beam specimen without precracking, according to the following formula. The dimensions of the beam specimen without precracking shall be the same as that of the specimen for fracture toughness test.

$$R = \frac{P}{T - D} \quad (\text{B.1})$$

where

R is the rigidity of entire testing system in newtons per metre;

P is the compression load applied to the bending test fixture in newtons (recommended to be in the range 100 N to 500 N);

T is the cross head displacement (stroke) of testing machine in metres (measured with built-in measurement equipment such as the linear variable differential transformer or the rotary encoder);

D is the elastic bending loading point deflection of the beam specimen in metres.

The elastic bending loading point deflection of the beam specimen, D , shall be calculated with the following formula.

$$D = \frac{P(d_1 - d_2)^2 [6d_2 + 2(d_1 - d_2)]}{8Edw^3} \quad (\text{B.2})$$

where

- d_1 is the distance between supporting roller pins in bend test fixture (lower span) in metres;
- d_2 is the distance between loading roller pins in four-point bend test fixture (upper span) (= 0 in three-point bend) in metres;
- E is the modulus of elasticity of the beam specimen (Young modulus) in pascals;
- d is the thickness of beam specimen in metres;
- w is the width of beam specimen in metres.

—

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