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# Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for fracture toughness of monolithic ceramics at room temperature by chevron-notched beam (CNB) method

*Céramiques techniques — Méthode d'essai de ténacité à la rupture des céramiques monolithiques à température ambiante sur éprouvette entaillée en chevron*

Reference number
ISO 24370:2005(E)

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# Contents

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# Foreword

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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 24370 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 toughness of monolithic ceramics at room temperature by chevron-notched beam (CNB) method

## 1 Scope

This International Standard specifies a test method for determining the fracture toughness of monolithic ceramic materials at room temperature by the chevron-notched beam (CNB) method.

This International Standard is applicable to monolithic ceramics and whisker- or particulate-reinforced ceramics that are regarded as macroscopically homogeneous. It is not applicable to continuous-fibre reinforced ceramic composites.

This International Standard is usually applicable to ceramic materials with a fracture toughness less than about 12 MPa($m^{1/2}$). The test method is applicable to materials with a flat crack-growth resistance curve and may be applicable to materials with a rising crack-growth resistance curve (R-curve).

## 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 7500-1:2004, *Metallic materials — Verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Verification and calibration of the force-measuring system*

ISO 14704:2000, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for flexural strength of monolithic ceramics at room temperature*

## 3 Terms and definitions

For the purposes 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 (mode I) displacement

NOTE It is a function of applied force and test specimen size, geometry and crack length.

**3.2**
**fracture toughness**
generic term for measures of the resistance of extension of a crack

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**3.3**
**fracture toughness value**
$K_{I,CNB}$
value of crack-extension resistance, i.e. fracture toughness, as measured by the CNB method

NOTE The measured stress intensity factor corresponds to a crack-extension resistance of a stably-extending crack in a chevron-notched beam specimen. The measurement is performed to the operational procedure herein and satisfies all the validity requirements.

NOTE The definition, interpretation and measurement of $K_{I,CNB}$ assume a flat crack-growth resistance curve.

**3.4**
**critical stress intensity factor**
$K_{Ic}$
critical value of $K_I$ at which fracture occurs

## 4 Symbols

$l_0$ chevron tip dimension, CNB method (Figure 2)

$l_1$ chevron dimension, CNB method, [$l_1 = (l_{11} + l_{12})/2$]

$l_{11}$ chevron dimension, CNB method (Figure 2)

$l_{12}$ chevron dimension, CNB method (Figure 2)

$B$ test specimen thickness (Figure 2)

$K_I$ stress intensity factor, Mode I

$K_{Ic}$ critical stress intensity factor, Mode I

$K_{I,CNB}$ fracture toughness value, chevron-notched beam method

$S_o$ flexure fixture outer span

$S_i$ flexure fixture inner span

$L$ test specimen length

$F_{max}$ maximum force applied to the test specimen by the test machine and thereby recorded (Figure 5)

$F_{Tare}$ force applied to the test specimen by the upper fixture

$F$ total force applied to the test specimen ($F_{max} + F_{Tare}$). This value is used in calculation of $K_{I,CNB}$

$T$ notch thickness or kerf resulting from cutting of the chevron notch (Figure 2)

$W$ test specimen width (Figure 2)

$Y^*_{min}$ minimum value of the stress intensity factor coefficient $Y^*$


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## 5 Principle

This International Standard is intended to be used for material development, material comparison, quality assurance, characterization, reliability analysis and design data generation. The chevron-notched beam (CNB) method measures the fracture toughness value $K_{I,CNB}$ by fracturing a flexural specimen, that has a chevron notch (Figures 1 and 2). The specimen is fractured by four-point flexure. Force versus displacement, and backface strain or time are recorded in order to detect unstable fracture. The fracture toughness value $K_{I,CNB}$ is calculated from the fracture load and the minimum stress intensity factor coefficient. Background information concerning this test method may be found in References [1] and [2]. An international interlaboratory comparison study (round robin) project on the chevron-notched method is described in Reference [3], and a comparison of this method to other standardized methods is given in References [2] and [4].

NOTE Ceramics generally exhibit stable crack extension from a chevron notch if the notch is sufficiently narrow ($<$ 0,30 mm), and the other notch dimensions are within the specified tolerances. If stable crack extension is not obtained, then the fracture toughness cannot be directly measured.

## 6 Apparatus

### 6.1 Test 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 7500-1:2004 Class 1, with an accuracy of 1 % of the indicated force at fracture.

### 6.2 Flexure fixtures

A schematic diagramme of a typical flexure fixture and test specimen is shown in Figure 1. Flexure fixtures shall meet the requirements of ISO 14704. The fixtures should be semi-articulating. Test specimens shall be contacted by smooth cylindrical bearings with a diameter between 4,50 mm and 5,00 mm. The diameter should be uniform to ± 0,015 mm.

The bearings shall be free to roll in order to minimize friction, and the two inner bearings shall be free to roll inward, and the two outer bearings shall be free to roll outward. The inner span, $S_i$, should measure 20 mm ± 0,5 mm and the outer span, $S_o$, should measure 40 mm ± 0,5 mm. Alternatively, the inner and outer span may measure 10 mm and 30 mm, respectively.

When specific test environments other than the laboratory air are employed, an adequate chamber to hold the environment around the test fixture is required. For gaseous environments such as dry nitrogen, a polyethylene bag can be used. For liquid environments such as silicone oil or water, the specimen can be coated and placed in the fixture or the fixture and test specimen can be immersed in a chamber containing the liquid.

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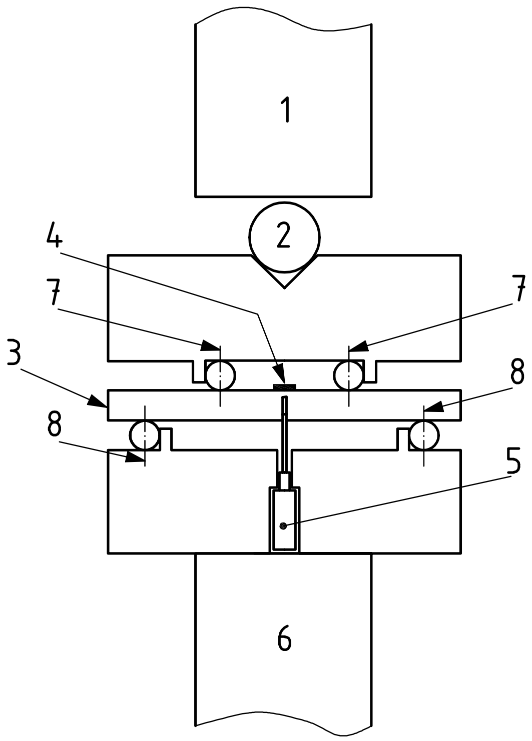


**Key**

1 push rod
2 ball
3 test specimen
4 strain gauge
5 displacement transducer
6 support rod
7 flexure fixture inner span, $S_i$
8 flexure fixture outer span, $S_o$

**Figure 1 — Schematic example of four-point flexure of a chevron-notched test specimen**

## 6.3 Micrometer

A micrometer such as shown in ISO 3611[11] but with a resolution of 0,002 mm shall be used to measure the test specimen dimensions. The micrometer shall have flat anvil faces such as shown in ISO 3611[11]. The micrometer shall not have a ball tip or sharp tip since these might damage the specimen. Alternative dimension-measuring instruments may be used provided that they have a resolution of 0,002 mm or finer.

## 6.4 Optical microscope

A travelling microscope or an optical microscope equipped with a calibrated filar eyepiece should be used to measure chevron notch dimensions $l_0$, $l_{11}$, $l_{12}$ and $T$. Magnifications of 10 × to 50 × are usually required. The dimensional measurement performance of the measurement system shall be calibrated with a reference standard.


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### 6.5 Stability detection equipment

The stability of the test is detected by monitoring the test specimen centre-point displacement, load-point displacement, actuator displacement, cross-head displacement or backface strain. Alternatively, force can be recorded as a function of time. Examples of force as a function of strain, actuator stroke and time are shown in Figure 3.

Both backface strain and extensometers placed within or near the flexure fixture are excellent for detecting the stability of the test [5] [6] [7]. Test system extensometers that are placed remotely relative to the test specimen are less sensitive to the local events in the test specimen and may not detect stable extension. Monitoring force as a function of time is a less effective method of detecting stable crack extension. This is particularly the case for materials with a low fracture toughness [e.g. $< 3{,}0$ MPa($m^{1/2}$)] and high elastic modulus (e.g. 400 GPa). Reference [2] discusses experience with various monitoring methods.

If an extensometer contacting the test specimen is used, the force of the extensometer on the specimen should be less than 0,2 N.

## 7 Test specimens

### 7.1 Geometry, size, preparation and edge chamfering

#### 7.1.1 Recommended geometry

Rectangular beams with dimensions shown in Figure 2 should be used. Cross-sectional tolerances should be ± 0,20 mm.

The parallelism tolerance on opposite longitudinal faces should be 0,015 mm. The test specimen illustrated in Figure 2 resulted in excellent correlation to other standardized test methods for a wide variety of ceramics [2], and has been used in the development of a reference material [4]. The stress intensity factor coefficient is based on the straight-through-crack-assumption model and correlates well with finite element analysis (FEA) models for the range allowed [2].

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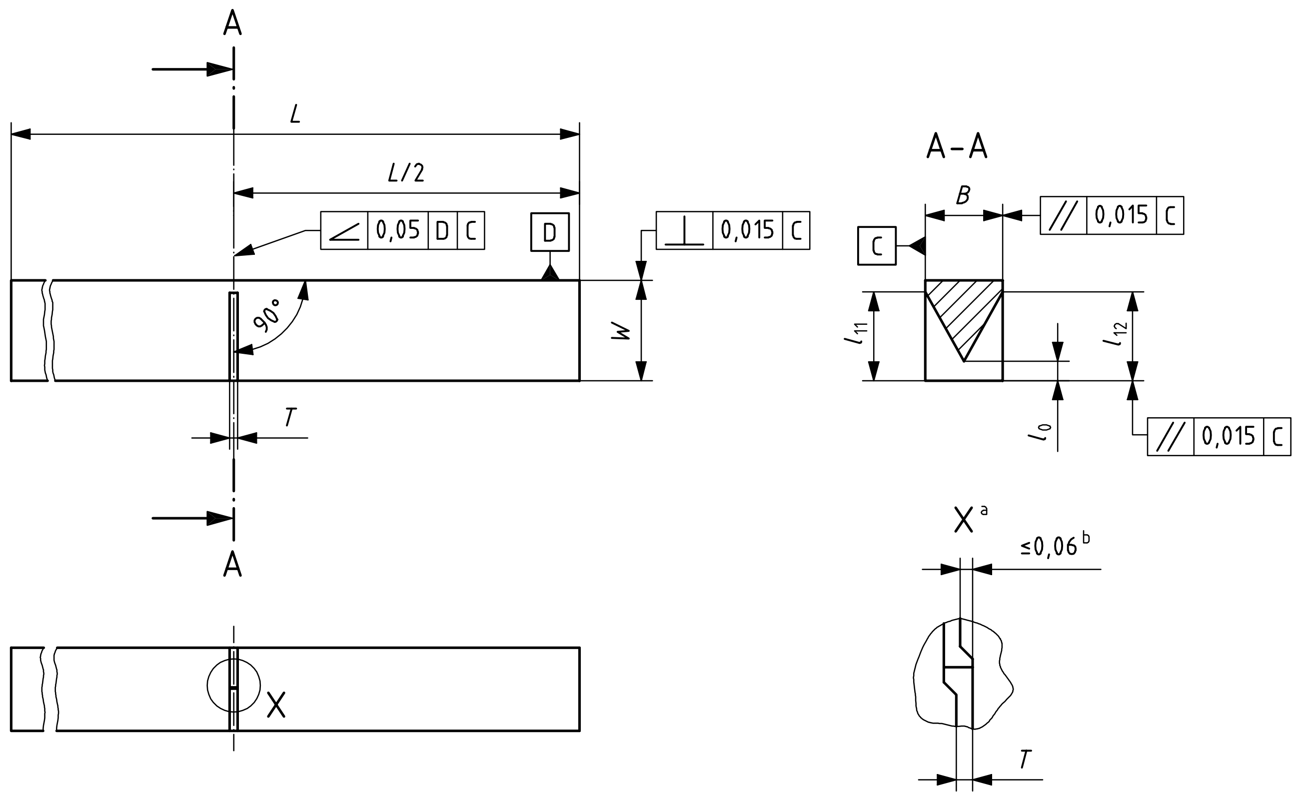
Dimension in millimetres




**Key**

| | |
|---|---|
| $L$ | test specimen length, min. 45 |
| $L/2$ | 22,5 ± 0,50 |
| $B$ | test specimen thickness, 3,00 ± 0,20 |
| $W$ | test specimen width, 4,00 ± 0,20 |
| $l_0$ | chevron tip dimension, CNB method, 0,80 ± 0,08 |
| $l_{11}$ | chevron dimension, CNB method, min. 3,80, max. $W$; no overcut |
| $l_{12}$ | chevron dimension, CNB method, min. 3,80, max. $W$; no overcut |
| $T$ | notch thickness or kerf, max. 0,30 |

Do not bevel edges.

Notch planes should meet within 0,06 mm.

a Notch tip detail.

b Chevron tip on centreline within 0,06 mm.

**Figure 2 — Test specimen configuration and terminology for the recommended geometry**



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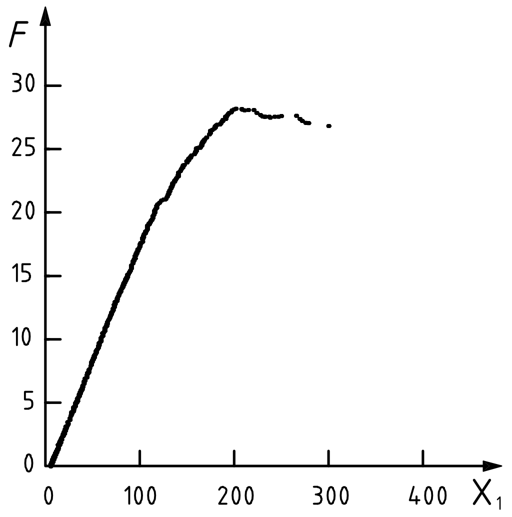
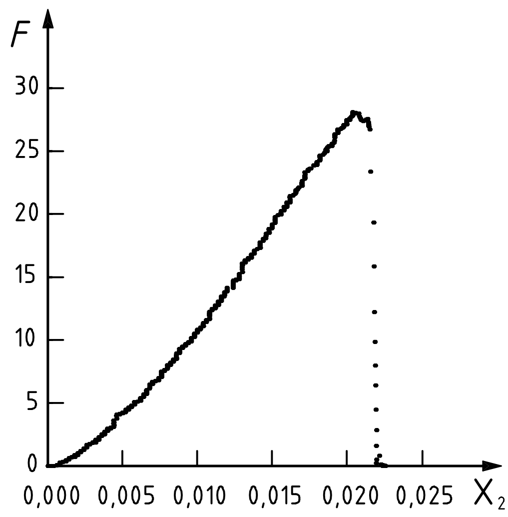
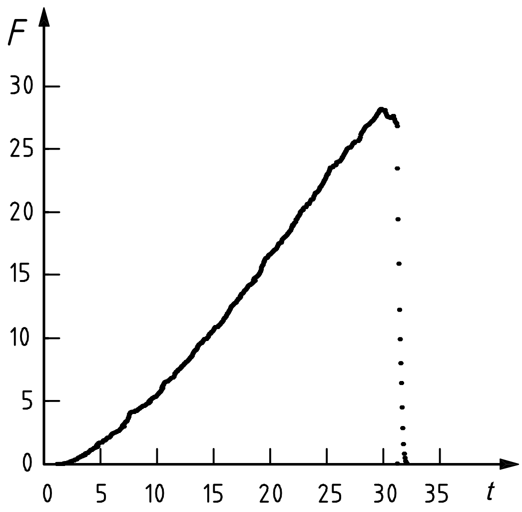



a) Force as a function of strain




b) Force as a function of actuator stroke




c) Force as a function of time

**Key**

$X_1$ back-face strain ($\times 10^6$)

$X_2$ actuator stroke, millimetres

$t$ time, seconds

$F$ force, newtons

NOTE 1 A 20 Hz data acquisition rate was used.

NOTE 2 Test material was alpha silicon carbide.

**Figure 3 — Examples of force as a function of strain, actuator stroke and time**

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### 7.1.2 Alternative geometry

In some instances, circumstances such as availability of test material, existing test configurations, machining capability, or other considerations may dictate a choice of test specimen geometry and/or dimensions other than those recommended in 7.1.1. In those instances, alternative geometries and basic dimensions (e.g. $L$, $W$, $B$) can generally be employed with the following guidelines. Basic dimensions and tolerances should be proportional to those recommended. In addition, values of $\alpha_1 = l_1/W$ and $\alpha_0 = l_0/W$ should be chosen to be in the ranges of $\alpha_1 = 0{,}9$ to 1,0 and $\alpha_0 = 0{,}12$ to 0,24. The stress intensity for the alternative geometry given in 9.1.2 shall be used. A drawing of the alternative test specimen geometry and test configuration shall be included in the test report.

### 7.1.3 Preparing test specimens and chamfering

Test specimens shall be prepared in accordance with ISO 14704 with an exception: the specimen edges should not be chamfered or bevelled. The presence of a chamfer or bevel can interfere with measurement of the chevron-notched depth.

### 7.1.4 Cutting the chevron notch

Cut the chevron notch using a D76 grit (equivalent to 220 grit in the U.S. designation and corresponding to a nominal grain diameter of $>$ 63 µm) or finer diamond-grit wheel at a rate of not more than 0,002 mm per pass for the final 0,06 mm. The notch thickness, $T$, should be less than 0,30 mm at any point of its intersection with the test specimen surface. (See also the requirements in Figure 2.) Planes of notches cut from each side of the test specimen shall meet within 0,2 $T$. The tip of the chevron shall be on centre within 0,02 $B$.

NOTE 1 Grit size designations for diamond wheels can be found in ISO 6106 [12].

NOTE 2 The use of specifically-designed machining fixtures for producing the chevron notch has been shown to reduce machining costs and increase the consistency of the chevron notch [8].

NOTE 3 Larger notch thickness is acceptable provided that stable crack extension occurs. Using a V-shaped notch (which has a larger notch thickness where it intersects the specimen surface than at the root of the notch), rather than a straight notch thickness, resulted in more consistent fracture toughness measurements [3] [9]. Generally, notch thicknesses between 0,25 mm and 0,30 mm at any point on the specimen surface are available from commercial machine shops. The offset between the notch planes is particularly critical.

— For alpha silicon carbide, notch thicknesses of 0,225 mm to 0,250 mm with an offset less than 0,030 mm were consistently stable.

— For sapphire, notch thicknesses of 0,300 mm to 0,325 mm with notch offsets of 0,075 mm to 0,110 mm were consistently unstable. However, notch thicknesses of 0,250 to 0,300 mm with offsets of 0,025 mm to 0,075 mm were stable. Wider notches may allow for more total offset.

Residual stresses introduced by the machining process can lead to inconsistent crack initiation and subsequent unstable crack propagation. If such behaviour is observed, a simple compression-compression procedure may be used to damage the chevron tip and thereby promote stable crack initiation.

— Place the test specimen upside down in the test fixture and load it several times to approximately three times the expected fracture load for testing in the normal position.

— After this compression loading, remove the test specimen and test it as specified in Clause 8.

## 7.2 Number of specimens

The number of valid tests shall be not less than five. It is recommended that at least ten test specimens be prepared. This will provide extra test specimens to determine if stable crack growth can be attained without extra preparation. It will also provide test specimens to make up for unsuccessful or invalid tests. More test specimens are needed if the environment is to be varied.


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# 8 Procedure

## 8.1 Permitted test environments

### 8.1.1 Laboratory ambient environment

Fracture the test specimens in laboratory ambient conditions. Record the test temperature and relative humidity.

NOTE Many oxides, glasses and ceramics having glassy boundary phases can be susceptible to slow crack growth. Measured fracture toughness can be sensitive to test rate and moisture in the atmosphere.

### 8.1.2 Application-specific environment

Fracture the test specimens in the desired environment (e.g. distilled water). Record the test temperature and relevant information regarding the environment.

### 8.1.3 Inert environment

Use an inert environment such as dry nitrogen gas or silicone oil. Select an atmosphere that is considered not to adversely affect the crack growth of the test specimen during the test. Recommended environments include dry air, nitrogen or argon with purity of 99,9 % or better at atmospheric pressure, vacuum of less than 0,13 Pa, or silicone or paraffin oil, preferably with low water content. If silicone oil is used, bake the test specimens and the oil separately at approximately 110 °C for one hour prior to covering the test specimens with the oil. It is advised that the oil be poured over the test specimen, because dropping them into a container of oil may induce fracture.

NOTE For inert atmosphere testing, a simple chamber around the test fixtures or even a sealed plastic bag may adequate, provided that the laboratory ambient air can be flushed from the chamber for several minutes between tests.

## 8.2 Test specimen dimensions and alignment

### 8.2.1 Test specimen dimensions

Measure and record the test specimen dimensions, $B$ and $W$, in the vicinity of the chevron notch to within 0,002 mm.

NOTE Some differences exist in the definitions and symbols for “thickness” and “width” between strength tests and fracture mechanics tests.

— In fracture mechanics tests, the horizontal dimension $B$ in Figure 2, is referred to as the thickness, and the vertical dimension, $W$, is referred to as the width.

— In strength testing, $B$ is the vertical dimension and referred to as the height, depth, or thickness and $W$ is the horizontal dimension when the specimen is in the usual test orientation.

— The fracture mechanics definitions may have emerged from the testing of metallic sheets and plates, and the need for a minimum thickness to obtain plane–strain conditions.

### 8.2.2 Measure the chevron tip dimension, chevron dimensions and notch thickness

Measure the chevron tip dimension, $l_0$, from the chevron tip to the specimen surface at the notch mouth (i.e. opposite the tip of the chevron) to 0,03 mm. Alternatively, $l_0$ may be measured after the test. Measure the chevron dimensions, $l_{11}$ and $l_{12}$, where the notch groove meets the specimen surface and calculate $l_1$, the average of the two values. The difference between the average and the individual values shall be no more than 0,02 $W$. Measure the notch thickness $T$ within 0,03 mm. The notch thickness should be less than 0,30 mm.

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### 8.2.3 Measure the inner and outer span

Measure the inner and outer spans to 0,06 mm. If the upper fixture rests freely on the test specimen and thereby places a tare force on the test specimen, measure the weight of the upper fixture and its components (e.g. bearing ball, etc.). This force, $F_{Tare}$, will be added to the relevant maximum test force, $F_{max}$, to determine the total force, $F$, applied to the test specimen at fracture.

### 8.2.4 General guidance on bend testing

For general guidance on bend testing, see ISO 14704. It is recommended that only semi-articulated fixtures be used.

- Insert the test specimen into the flexure fixture with the chevron tip on the tension face, as shown in Figure 1.
- Align the chevron notch within 0,5 mm of the midpoint between the two inner rollers, $S_i$, of the four-point flexure fixture.
- Align the test specimen laterally within the test fixture so that the specimen is directly below the axis of applied load within 0,1 mm.
- The test specimen should be preloaded to no more than 25 % of the expected fracture force.

### 8.2.5 Fracturing the test specimen

Apply an increasing compressive force to the fixture until the test specimen fractures. A displacement rate of 0,05 mm/min is recommended; however, rates of 0,02 mm/min to 0,2 mm/min may be used. Measure the maximum fracture force, $F_{max}$, to an accuracy of ± 1 %.

## 8.3 Post-test measurements

Examine the chevron notch at sufficient magnification (~30 ×). The tip of the chevron shall be on centre within 0,02 $B$.

## 8.4 Post-test interpretation

The test record shall exhibit a smooth, nonlinear transition to the maximum force prior to final fracture as shown in Figure 3. If the test specimen exhibits a sudden drop in force that is not followed by a subsequent nonlinear force increase, the test is unstable and invalid (See Figure 4). Determine the relevant maximum test force, $F_{max}$, from the test record. In some cases the test specimen will overload slightly or pop-in at crack initiation, as shown in Figure 5. In the calculations, use the relevant maximum test force associated with stable crack extension, as marked in Figure 5.


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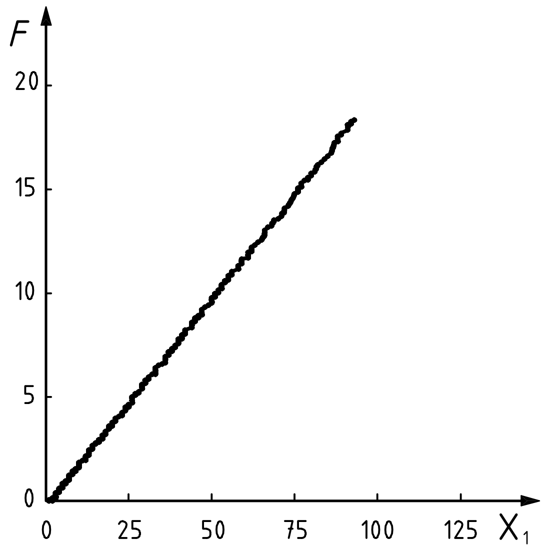
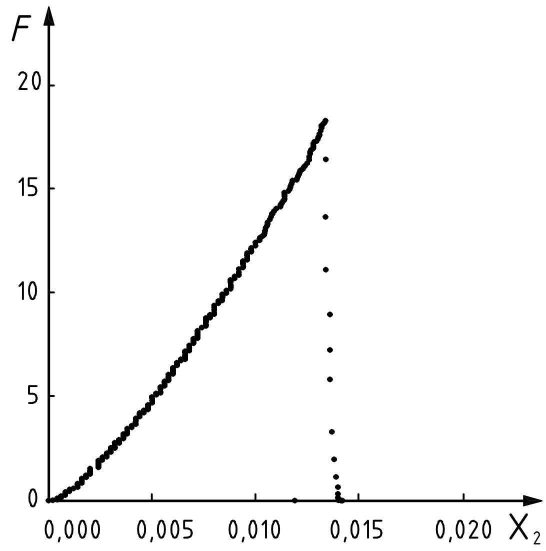
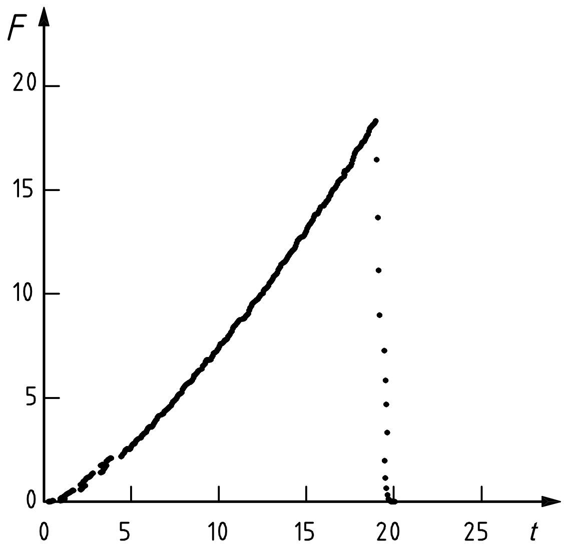



**a) Force as a function of strain**




**b) Force as a function of actuator stroke**




**c) Force as a function of time**

**Key**

$X_1$ back-face strain ($\times 10^6$)

$X_2$ actuator stroke, millimetres

$t$ time, seconds

$F$ force, newtons

NOTE 1 A 20 Hz data acquisition rate was used.

NOTE 2 Test material was alpha silicon carbide.

**Figure 4 — Examples of unstable fracture as a function of strain, actuator stroke and time**

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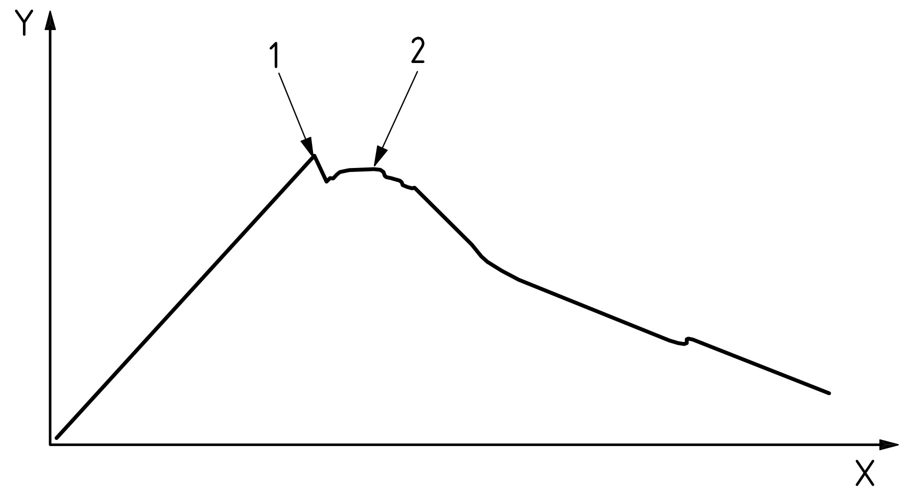


**Key**

X displacement
Y force

1 pop-in
2 maximum force applied to a test specimen by the test machine and thereby recorded, $F_{max}$

**Figure 5 — Example of an unstable crack jump followed by stable fracture**

## 9 Calculation

### 9.1 Calculations of the minimum stress intensity factor coefficient $Y^*_{min}$

#### 9.1.1 Recommended test specimen geometry

Calculate the minimum stress intensity factor coefficient $Y^*_{min}$ from the following function [2].

$$Y^*_{min}\left(l_0/W, l_1/W\right) = \frac{0{,}387\,4 - 3{,}091\,9\,(l_0/W) + 4{,}201\,7\,(l_1/W) - 2{,}312\,7\,(l_1/W)^2 + 0{,}637\,9\,(l_1/W)^3}{1{,}000 - 2{,}968\,6\,(l_0/W) + 3{,}505\,6\,(l_0/W)^2 - 2{,}137\,4\,(l_0/W)^3 + 0{,}013\,(l_1/W)} \quad (1)$$

The function is accurate to better than 0,5 % for

— $0{,}175 < l_0/W < 0{,}225$ and

— $0{,}950 < l_1/W < 1{,}000$.

EXAMPLE:

If the following values are used

$W = 4{,}00$ mm $= 4{,}00 \times 10^{-3}$ m,

$l_0 = 0{,}80 \times 10^{-3}$ m, and

$l_1 = 3{,}89$ mm $= 3{,}89 \times 10^{-3}$ m


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then

$l_0/W = 0{,}20$,

$l_1/W = 0{,}973$, and

$Y^*_{min} = 4{,}16$.

### 9.1.2 Alternative test specimen geometry

For an alternative test specimen geometry with notch dimensions within $0{,}12 < l_0/W < 0{,}24$ and $0{,}90 < l_1/W < 1{,}00$, calculate the minimum stress intensity factor coefficient $Y^*_{min}$ from the following function.

$$Y^*_{min}\left(l_0/W, l_1/W\right) = \left(2{,}92 + 4{,}52\left(l_0/W\right)\frac{l_0}{W} + 10{,}14\left(l_0/W\right)^2\right)\sqrt{\frac{\left(l_1/W\right) - \left(l_0/W\right)}{1{,}000 - \left(l_0/W\right)}} \quad (2)$$

The function is accurate to better than 3 % for

— $0{,}12 < l_0/W < 0{,}24$ and

— $0{,}90 < l_1/W < 1{,}00$. [10]

## 9.2 Calculation of the fracture toughness value, $K_{I,CNB}$

Calculate the fracture toughness value, $K_{I,CNB}$, from the following equation:

$$K_{I,CNB} = \frac{F\left(S_o - S_i\right)}{BW^{3/2}} \times \frac{Y^*_{min}}{\sqrt{1000}} \quad (3)$$

where

| | |
|---|---|
| $K_{I,CNB}$ | is the fracture toughness value, in MPa($m^{1/2}$) or MN($m^{-3/2}$); |
| $F$ | is the total force, in newtons (maximum force, $F_{max}$, plus tare force, $F_{Tare}$); |
| $S_o$ | is the outer span, in millimetres; |
| $S_i$ | is the inner span, in millimetres; |
| $B$ | is the test specimen thickness, in millimetres (see Figure 2); |
| $W$ | is the test specimen width, in millimetres (see Figure 2); |
| $Y^*_{min}$ | is the stress intensity factor coefficient (dimensionless). |

## 10 Test report

The test report shall include the following information:

a) test specimen identification;

b) form of product tested (e.g. sintered, hot-pressed), if data are available;

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c) crack plane orientation, if available;

d) environment of test, relative humidity, temperature;

e) test specimen dimensions $B$ and $W$;

f) notch dimensions, $l_0/W$, $l_{11}/W$, $l_{12}/W$ and $T$.

Include statements about the validity of the chevron notches and their dimensions (8.3 and 8.4);

g) declaration of whether the recommended or an alternative test specimen geometry/configuration was used.

If an alternative test specimen geometry/configuration was used, include a dimensioned drawing of the test specimen and test fixture;

h) test fixture tare force, $F_{\text{Tare}}$;

i) method used to detect stable crack extension;

j) displacement rate;

k) number of valid tests;

l) individual fracture toughness values, $K_{\text{I,CNB}}$;

m) mean fracture toughness and standard deviation;

n) each load plot, with a statement about stability (8.4).


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