INTERNATIONAL STANDARD

ISO 18608

First edition
2017-03

Fine ceramics (advanced ceramics, advanced technical ceramics) — Mechanical properties of ceramic composites at ambient temperature in air atmospheric pressure — Determination of the resistance to crack propagation by notch sensitivity testing

Céramiques techniques — Propriétés mécaniques des céramiques composites à température ambiante sous pression atmosphérique — Détermination de la résistance à la propagation de fissure par un essai de sensibilité à l’entaille

Reference number
ISO 18608:2017(E)

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Foreword

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

Fine ceramics (advanced ceramics, advanced technical ceramics) — Mechanical properties of ceramic composites at ambient temperature in air atmospheric pressure — Determination of the resistance to crack propagation by notch sensitivity testing

1 Scope

This document describes a method for the classification of ceramic matrix composite (CMC) materials with respect to their sensitivity to crack propagation using tensile tests on notched specimens with different notch depths. Two classes of ceramic matrix composite materials can be distinguished: materials whose strength is sensitive to the presence of notches and materials whose strength is not affected. For sensitive materials, this document defines a method for determining equivalent fracture toughness.

The parameter, K_{eq}, is defined as the fracture toughness of a homogeneous material which presents the same sensitivity to crack propagation as the ceramic matrix composite material which is being considered. The definition of the K_{eq} parameter offers the possibility to compare ceramic matrix composite materials with other materials with respect to sensitivity to crack propagation.

For notch insensitive materials, the concept of K_{eq} does not apply.

This document applies to all ceramic matrix composites with a continuous fibre reinforcement, unidirectional (1 D), bidirectional (2 D), and tridirectional (x D, where $2 < x \leq 3$), loaded along one principal axis of reinforcement.

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 3611, *Geometrical product specifications (GPS) — Dimensional measuring equipment: Micrometers for external measurements — Design and metrological characteristics*

ISO 7500-1, *Metallic materials — Calibration and verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Calibration and verification of the force-measuring system*

ISO 15733:2015, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Mechanical properties of ceramic composites at ambient temperature in air atmospheric pressure — Determination of tensile properties*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 15733 and the following apply.

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

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

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

3.1
ligament
part of the double edge notched specimen that is located between the notches

Note 1 to entry: The width of the ligament is denoted b; the cross-section of the ligament is denoted A.

3.2
notch depth
a
distance between the side of the specimen and the tip of the notch

3.3
notched specimen width
b_n
width of the notched specimen outside the notched cross-section

3.4
maximum tensile force
F_m
highest recorded tensile force on the test specimen when tested to failure

3.5
un-notched specimen tensile strength
$\sigma_{t,m}$
tensile strength determined by measurement according to ISO 15733

Note 1 to entry: The value of this parameter is designated σ_r.

3.6
notched specimen tensile strength
$\sigma_{t,m,n}$
ratio of the maximum tensile force to the ligament cross-section area

Note 1 to entry: The value of this parameter is designated σ_n.

3.7
equivalent fracture toughness
K_{eq}
fracture toughness of a homogeneous and isotropic material which presents the same dependence of the stress ratio σ_n/σ_r on the notch depth as the investigated composite

4 Principle

Tensile tests are carried out on double edge notched test specimens with notches of different depths. The results of these tests are compared with the results of tensile tests on specimens without notches. The cross-sectional dimensions of the notched specimens between the notches are equal to those of the un-notched specimens.

The strength values observed on both types of specimens as a function of notch depth allow the determination of the range of notch size for which the tested composite is sensitive to the presence of notches.

5 Significance and use

The fracture toughness is a material property which characterizes the initiation of fracture from a sharp crack (usually obtained by fatigue cracking under plane strain conditions). The fracture toughness of materials at the onset of crack extension from a pre-existing fatigue crack is characterized by the value of one of the following parameters:

a) K_{Ic}, a critical value of K_I (the stress intensity factor of the elastic stress field in the vicinity of the crack front) at the point of instability of the crack extension;

b) G_{Ic}, a critical value of G_I (the strain-energy release rate with crack extension per unit area of newly created crack surface) at the point of instability of the crack extension;

c) J_{Ic}, a critical value of J_I (a line or surface integral used to characterize the local stress-strain field around the crack front) at the onset of stable crack extension.

The J integral plays an important role in nonlinear fracture mechanics. It applies to nonlinear elastic bodies, whereas linear elastic fracture mechanics (K_{Ic} and G_{Ic}) consider linear elastic bodies.

Several problems arise in determining and even in defining K_{Ic}, G_{Ic} and J_{Ic} in fibre reinforced ceramic matrix composites, as a result of the following features:

a) CMC are generally highly heterogeneous, consisting of different constituents (fibres and matrix), and containing pores and cracks;

b) in some CMC, a damage zone of multiple matrix cracks forms ahead of a notch prior to ultimate failure;

c) the associated deformations are nonlinear.

The load versus load line displacement curve from a fracture test on a notched specimen involves a nonlinear domain induced by diffuse micro-cracking within the matrix at the notch tip. The damage zone is in the millimetre to centimetre scale (from one to several tow diameters). At maximum load, a macroscopic crack is created from the random failure of fibres within those tows located in the damage zone. Crack extension in CMC, hence, does not result from the mechanism of extension of a single macroscopic crack as observed in monolithic materials.

Because of the presence of the damage zone and of heterogeneous microstructure, the stress distribution in the damage zone differs from the one induced ahead of the crack tip in linear elastic bodies. The K_I parameter does not describe the stress field in the region ahead of the crack tip. A critical value K_{Ic} cannot be defined.

The main difficulty in the determination of the strain energy release rate G_I, as well as the J integral, results from the presence of the micro-cracked zone at the notch tip (which is not small compared with the specimen dimensions) and the jagged surface of the macroscopic crack. As a consequence, an increase in crack length can neither be easily defined nor measured.

Tensile tests performed on specimens containing holes or notches have demonstrated that many CMC are relatively notch-insensitive over a range of notch sizes. The net-section stress at fracture is typically (80 to 100) % of the un-notched strength. Notch insensitivity results from a stress relaxation at the notch tip due to the development of the damage zone. As a consequence, the fibres in the damage zone are subjected to stresses that are comparable in magnitude to the remote stresses.

A measure of the notch sensitivity at a given notch depth is provided by the ratio of the failure stress of a notched tensile specimen (σ_n) to the failure stress of a corresponding un-notched tensile specimen (σ_r):

a) when $\sigma_n < \sigma_r$, the composite is notch sensitive;

b) when $\sigma_n \geq \sigma_r$, the composite is notch insensitive.

The stress ratio σ_n/σ_r is a useful parameter for component design purposes. It allows the selection of the composites that are able to tolerate notches, holes, etc.

For material comparison purposes, an equivalent fracture toughness K_{eq} is defined over the notch depth range where the stress ratio is less than 1.

K_{eq} represents the fracture toughness of the equivalent homogeneous monolithic material which exhibits the same notch sensitivity as the actual composite. K_{eq} is calculated from the dependence of the σ_n/σ_r stress ratio on notch depth, using linear elastic fracture mechanics equations.

Over the range of notch depths where the CMC is notch sensitive, the calculation of the equivalent fracture toughness for the different notch depths does not usually result in a single value for K_{eq}. For reasons of conservatism, the minimum value is used.

For some CMC, a transition from notch insensitive to notch sensitive has been observed with increasing notch depth. The determination of equivalent fracture toughness is not recommended when the notch insensitive range extends beyond a minimum value of notch depth (1 mm).

Additional testing at different notch depths may be performed to provide a more complete understanding of the notch depth range where the CMC is notch insensitive.

6 Apparatus

6.1 Test machine

The machine shall be equipped with a system for measuring the force applied to the test specimen, which, when tested in accordance with ISO 7500-1, shall meet the requirements of grade 1 or better of that standard.

6.2 Load train

6.2.1 General

The load train is composed of the moveable and fixed crosshead, the loading rods, and the grips. Load train couplers may additionally be used to connect the grips to the loading rods.

The load train shall align the specimen axis with the direction of load application without introducing bending or torsion in the specimen. The alignment shall be verified and documented. The maximum per cent bending shall not exceed 5 at an axial strain of 500×10^{-6}.

6.2.2 Grips

The grips transmit the axial load applied by the testing machine to the specimen. They shall prevent slipping of the specimen in the gripping section. The selection of a particular type of grips depends on the specimen design and critically influences the alignment.

When the grip design relies on friction to transmit the axial load to the specimen, the use of an adjustable clamping pressure is recommended.

Care should be taken to avoid the introduction of torsional loading on the specimen when tightening the grips.

6.2.3 Load train couplers

Load train couplers may be used to connect the grips to the loading rods. Their primary function is to assure axial alignment of the grips in the loading train.

NOTE 1 Load train couplers are of two types: fixed or non-fixed. Fixed couplers usually consist of angularity and/or concentricity adjusters. Non-fixed couplers promote self-alignment of the load train upon movement of the cross-head. This self-aligning action is limited by the inherent friction between moving parts of the couplers.

NOTE 2 The self-aligning action of non-fixed load train couplers may result in non-uniform loading of the unbroken ligament of the specimen after appearance of damage in the specimen, which can modify the shape of the tensile curve.

NOTE 3 The use of well-aligned couplers and grips does not guarantee low bending in the specimen. The latter additionally depends on the type and operation of the grips, and on the type of specimen.

6.3 Data recording system

A calibrated recorder may be used to record the force-deformation curve. However, the use of a digital data recording system combined with an analogue recorder is recommended.

6.4 Micrometers

Micrometers used for the measurement of the dimensions of the test specimen shall be in accordance with ISO 3611.

6.5 Ligament size measuring device

A profile projector or any other suitable instrument shall be used to measure the width of the ligament between notches.

7 Specimens

7.1 Un-notched test specimens

All flat specimens from ISO 15733 can be used except type 2.

7.2 Notched test specimens

These shall be flat and shall have the same ligament cross-sectional dimensions and total length as the un-notched specimens (see Figure 1).

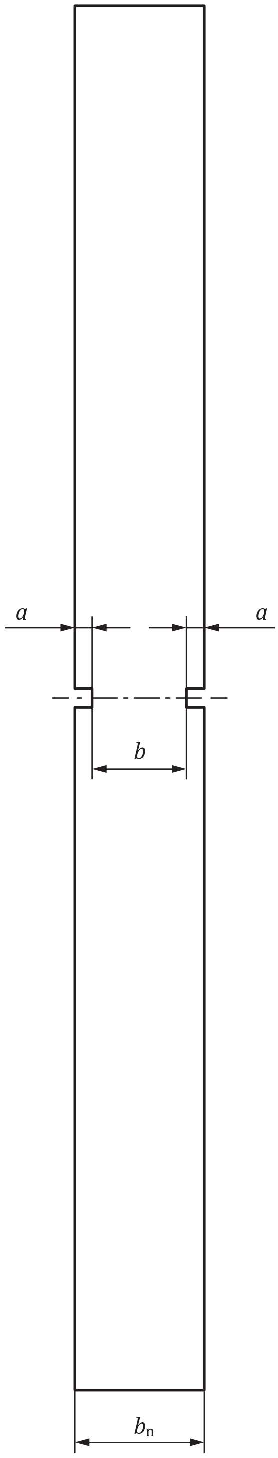

Figure 1 — Notched test specimens, with dimensions a, b and b_n as defined in Clause 3

7.3 Notches

Two identical notches shall be located at the centre on opposite sides of the test specimens in the same plane perpendicular to the load axis with a maximum width of 0,5 mm and a maximum deviation from this plane of 0,1 mm.

NOTE Notches can be prepared by different means (grinding, sawing, etc.). For the application of this document, the shape of the notch root is not critical.

At least four different notch depths shall be used, as set out in Table 1.

The minimum value of the notch depth shall be 1 mm.

The tolerance on *a* and *b* shall be +0,1 mm.

Table 1 — Notch dimensions

	Ligament width, *b* (mm)	Notch depth, *a* (mm)
Reference	≥8	0
1st series	≥8	0,1*b*[a]
2nd series	≥8	0,2*b*
3rd series	≥8	0,3*b*
4th series	≥8	0,5*b*

[a] If 0,1*b* is less than 1 mm, a notch depth *a* of 1 mm should be used.

8 Test specimen preparation

8.1 Machining and preparation

During cutting out, care shall be taken to align the test specimen axis with the desired fibre related loading axis.

Machining parameters which avoid damage to the material shall be established and documented. These parameters shall be adhered to during test specimen preparation.

8.2 Number of test specimens

At least three valid test results for each notch depth, as specified in 9.2.3, are required.

9 Test procedure

9.1 Test on reference specimen

Tests on reference specimens shall be carried out according to the procedure described in ISO 15733.

9.2 Test on notched specimen

9.2.1 Displacement rate

For tests on notched specimens, use the same displacement rate as for the tests on the reference specimens as specified in ISO 15733.

9.2.2 Measurement of test specimen dimensions

The width and the thickness of the specimens shall be measured to an accuracy of ±0,01 mm.

The depth of each notch and the ligament dimensions shall be determined with an accuracy of ±0,01 mm.

9.2.3 Test technique

Install the test specimen in the loading system with its longitudinal axis coinciding with that of the test machine.

Care shall be taken not to induce flexural or torsional loads when installing the test specimen in the loading system.

Place the specimen in the grips and carry out the test according to ISO 15733:2015, 8.3.

The distance between the grips shall be greater than 40 mm.

9.2.4 Test validity

The following circumstances invalidate a test:

— failure to specify and record test conditions;

— failure to meet specified test conditions;

— specimen slippage;

— rupture of the test specimen outside ligament between the two notches (except for the smallest notch depths).

10 Calculation of results

10.1 Test specimen origin

A diagram illustrating the reinforcement directions of the material with respect to the longitudinal axis of the specimen shall accompany the test results.

10.2 Tensile strength of un-notched specimen

The tensile strength shall be determined in accordance with ISO 15733.

10.3 Tensile strength of the notched specimen

The tensile strength of the notched specimen is determined for each test using Formula (1):

$$\sigma_{\mathrm{t,m,n}} = \frac{F_{\mathrm{m}}}{A} \tag{1}$$

where

$\sigma_{\mathrm{t,m,n}}$ is the tensile strength, in MPa;

F_{m} is the maximum tensile force, in N;

A is the ligament cross-sectional area, in mm^2.

10.4 Plotting of notch sensitivity diagram

Plot the diagram of $\bar{\sigma}_{\mathrm{t,m,n}} / \bar{\sigma}_{\mathrm{t,m}}$ versus $2a/b$ using Formula (2):

$$\bar{\sigma}_{\mathrm{t,m,n}} / \bar{\sigma}_{\mathrm{t,m}} = f(2a/b) \tag{2}$$

where

$\bar{\sigma}_{t,m,n}$ is the mean value of $\sigma_{t,m,n}$ for each notched specimen;

$\bar{\sigma}_{t,m}$ is the mean value of $\sigma_{t,m}$ for each un-notched specimen;

a is the notch depth, in mm;

b is the width of the ligament, in mm.

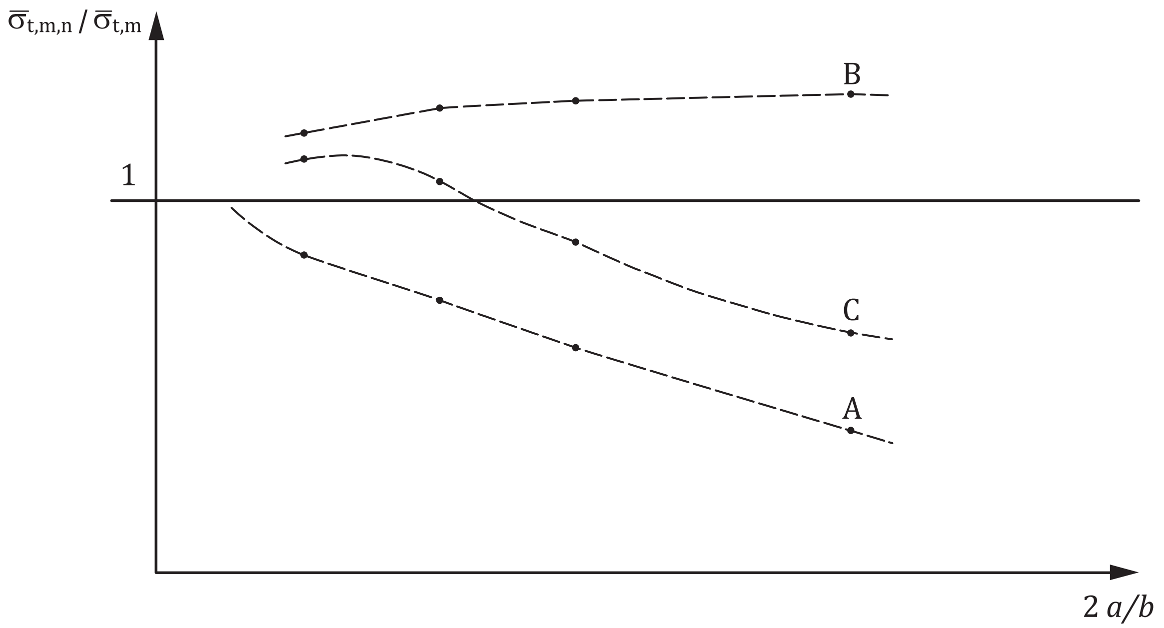

Figure 2 — Plot of $\bar{\sigma}_{t,m,n} / \bar{\sigma}_{t,m}$ versus 2*a*/*b*

From Figure 2, determine if the notch sensitivity is of class A, B, or C.

10.5 Calculation of equivalent fracture toughness for the different classes of behaviour

10.5.1 Class A behaviour

For each notch depth, calculate K_{eq} according to Formula (3):

$$K_{eq} = \sigma_{t,m,n} \sqrt{\pi a f(2a/b)} \tag{3}$$

where

K_{eq} is the equivalent fracture toughness, in MPa m½.

$$f(2a/b) = \left[1{,}122 - 0{,}56(2a/b) - 0{,}205(2a/b)^2 + 0{,}471(2a/b)^3 - 0{,}190(2a/b)^4\right] \tag{4}$$

When different values of K_{eq} are obtained with different notch depths, the smallest value is kept.

10.5.2 Class B behaviour

In this case, the concept of notch sensitivity does not apply.

10.5.3 Class C behaviour

In the notch-insensitive range, K_{eq} does not apply.

In the notch-sensitive range, a calculation of K_{eq} for comparison purposes is not recommended.

11 Test report

The test report shall contain the following information:

a) name and address of the testing establishment;

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

c) a reference to this document, i.e. ISO 18608;

d) test piece drawing or reference;

e) description of test material (material type, manufacturing code, batch number);

f) description of the test set-up (gripping system, load cell);

g) displacement rate;

h) number of tests carried out and the number of valid results obtained;

i) force-longitudinal deformation records;

j) valid results and mean values;

k) failure location of all the specimens;

l) classification of the material as sensitive or insensitive;

m) for a material which is notch sensitive, the value of the material equivalent fracture toughness;

n) for a material which is notch insensitive but which presents a transition towards notch sensitivity, the range of notch depths over which the material becomes sensitive.

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ICS 81.060.30

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