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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for flexural strength of monolithic ceramic thin plates at room temperature by three-point or four-point bending

Céramiques techniques — Méthode d'essai de la résistance en flexion des plaques minces en céramique monolithique à température ambiante en flexion trois ou quatre points

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for flexural strength of monolithic ceramic thin plates at room temperature by three-point or four-point bending

1 Scope

This document describes a test method for the flexural strength of monolithic ceramic thin plates at room temperature by three-point bending or four-point bending.

This document is intended for use with monolithic ceramics and whisker- or particulate-reinforced ceramics which are regarded as macroscopically homogeneous. It does not include continuous-fibre-reinforced ceramics composites. This document is applicable to ceramic thin plates with a thickness from 0,2 mm to 1,0 mm.

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

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 1101, *Geometrical product specifications (GPS) — Geometrical tolerancing — Tolerances of form, orientation, location and run-out*

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 14704:2016, *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.

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
flexural strength
maximum nominal stress at fracture of a specified elastic plate loaded in bending

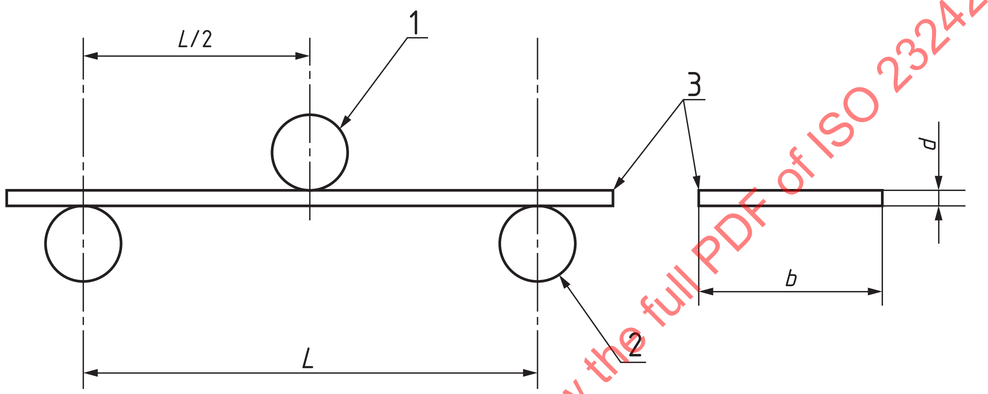
3.2
three-point flexure
configuration of flexural strength testing where a specimen is loaded at a location midway between two support bearings

Note 1 to entry: See Figure 1 a) and Table 1.

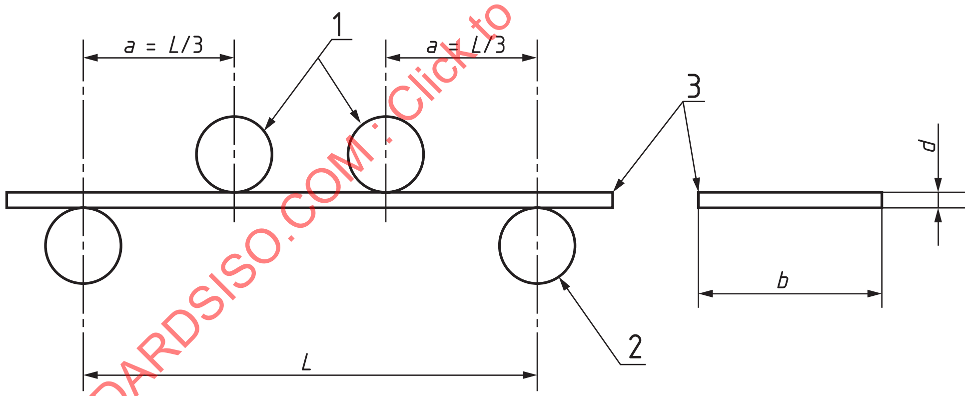
Note 2 to entry: The bearings may be cylindrical rollers or cylindrical bearings.

3.3
four-point flexure
configuration of flexural strength testing where a specimen is loaded equally by two bearings located symmetrically between two support bearings

Note 1 to entry: See Figure 1 b) and Table 1.



a) Three-point flexure



b) Four-point flexure

Key

1 loading bearing(s)
2 support bearings
3 specimen

Figure 1 — Flexural test configurations

Table 1 — Specimen type and dimension of bend test fixtures

Specimen type	Specimen thickness *d* mm	Bending mode	Lower span *L* mm	Length of fixture moment arm *a* mm	Diameter of bearings mm
I	0,5 ≤ *d* ≤ 1,0	3-point	30 ± 0,1	—	4,5 to 5,0
I	0,5 ≤ *d* ≤ 1,0	4-point	30 ± 0,1	10 ± 0,1	4,5 to 5,0
II	0,2 ≤ *d* < 0,5	3-point	15 ± 0,1	—	4,5 to 5,0
II	0,2 ≤ *d* < 0,5	4-point	15 ± 0,1	5 ± 0,1	2,5 to 3,0

3.4
fully articulating fixture
test fixture designed to apply uniform and even loading to test specimens that can have uneven, non-parallel or twisted surfaces

3.5
semi-articulating fixture
test fixture designed to apply uniform and even loading to specimens that have flat and parallel surfaces

4 Principle

A plate specimen with a rectangular cross-section is loaded in flexure until fracture. The load at fracture, the test fixture and specimen dimensions are used to compute the flexural strength of the specimen, which is a measure of the uniaxial tensile strength of a ceramic. The material is assumed to be isotropic and linearly elastic.

5 Apparatus

5.1 Bend testing machine

A suitable testing machine capable of applying a uniform crosshead speed shall be used. The testing machine shall be in accordance with ISO 7500-1, with an accuracy of 1 % of the indicated force at fracture. The test load of ceramic thin samples ranges roughly between 20 N and 200 N. A load cell shall be selected so that the fracture load of thin plates is not close to the limits of load capacity interval, i.e. the lower limit for high-load cell capacity and the upper limit for low-load cell capacity.

5.2 Bend test fixture

5.2.1 General

Three- or four-point flexure configurations shall be used, as illustrated in Figure 1 and Table 1. The fixture shall have bearings that are free to roll, as described in 5.2.2, in order to eliminate frictional constraints when the specimen surfaces expand or contract during loading. In addition, the fixture shall be designed so that parts “articulate” or tilt to ensure uniform loading to the specimen. The articulation shall be designed so that parts of the fixture can rotate, as specified in ISO 14704:2016, Figure B.1, to ensure even loading on the left and right bearings. An articulation is also needed to ensure that all the bearings contact the specimen surfaces evenly and apply a uniform load. Semi-articulated fixtures have some articulating or tilting capabilities and may be used with specimens that have flat and parallel surfaces, such as those of machined specimens. A semi-articulating fixture shall have pairs of upper and lower bearings that articulate to match the specimen surfaces, as specified in ISO 14704:2016, Figures B.2 and B.3. Fully articulated fixtures have more moving parts and shall be used for specimens that do not have flat and parallel surfaces. They allow independent articulation of the bearings. Fully articulated fixtures are often necessary for as-fired, heat-treated or oxidized specimens, since uneven loading can cause twisting and severe errors. A fully articulating fixture may also be used with machined specimens. For further detail on both semi-articulating and fully articulating fixtures, refer to ISO 14704.

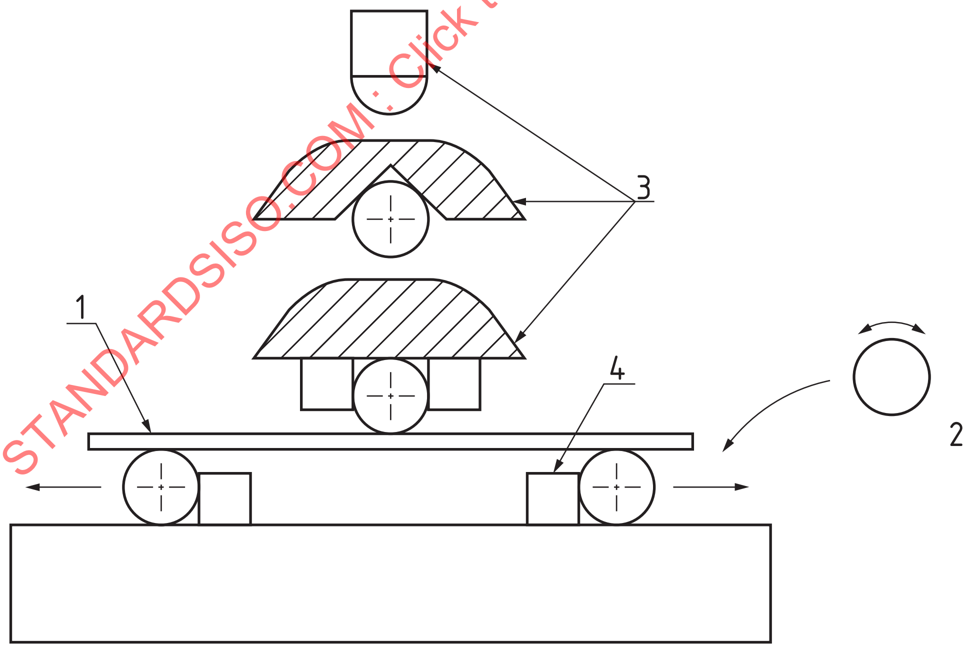
5.2.2 Bearings

Specimens shall be loaded and supported by bearings. The bearings may be cylindrical rollers or cylindrical pins. The bearings shall be made of a steel which has a hardness of no less than HRC 40 for specimen strengths up to 1 400 MPa, or no less than HRC 46 for specimen strengths up to 2 000 MPa. Alternatively, the bearing may be made of a ceramic or a hard metal with an elastic modulus between 200 GPa and 500 GPa and a flexural strength greater than 275 MPa. The bearing length shall be greater than or equal to 35 mm. The bearing diameter shall be between 4,5 mm and 5 mm, except that samples with a thickness of less than 0,5 mm are bent in a four-point bending. When samples with a thickness of less than 0,5 mm are bent in a four-point configuration, the bearing diameter shall be between 2,5 mm and 3 mm. The bearings shall have a smooth surface and shall have a diameter that is uniform to ± 0,015 mm. The bearings shall be free to roll in order to eliminate friction, as shown in Figure 2. In four-point flexure, the two inner bearings shall be free to roll inward, and the two outer bearings shall be free to roll outward. In three-point flexure, the two outer bearings shall be free to roll outward, and the inner (middle) bearing shall not roll.

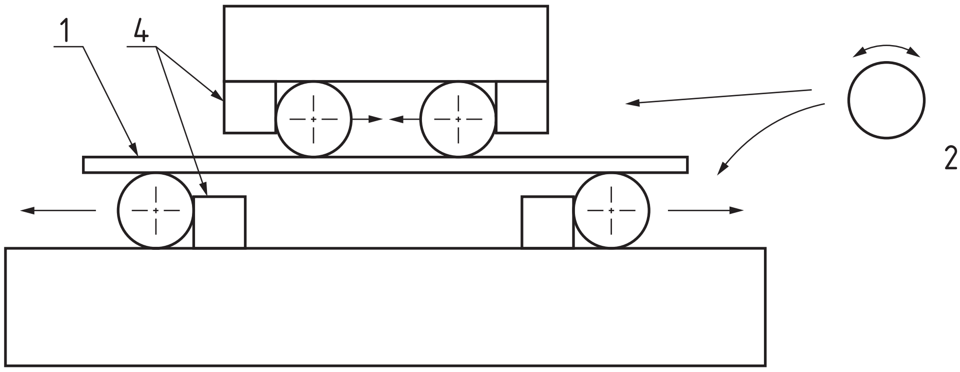
5.2.3 Positioning of bearings

Bearings shall be positioned so that their spans are accurate to within ± 0,1 mm. The middle bearing in a three-point fixture shall be positioned midway between the outer bearings to within ± 0,1 mm. The inner bearings in a four-point fixture shall be centred over the outer bearings to within ± 0,1 mm.

NOTE The positions of the bearings can be defined either by the use of captive bearings or by appropriate stops against which the bearings are held at the commencement of a test. The spans can be measured to the nearest 0,1 mm using a travelling microscope or other suitable device. The spans can also be verified by measuring the distances between bearing stops and adding (outer span) or subtracting (inner span) the radii of the bearing cylinders.



a) Three-point flexure



b) Four-point flexure

Key

1 specimen

2 alternative rolling bearings

3 alternative loading arrangements

4 stops

Figure 2 — Schematic illustration of fixtures showing rolling action of bearings and stops

5.2.4 Fixture material

The fixture which supports and aligns the bearings shall be of sufficient hardness to prevent the bearings from deforming the fixture permanently.

5.3 Calibrated micrometer

The calibrated micrometer shall be similar to one in accordance with ISO 3611 but capable of being read to a precision of 0,002 mm using a vernier or electronic readout. The micrometer shall have flat anvil faces. A micrometer with a ball tip or sharp tip shall not be employed due to its potential to introduce surface flaws into the specimen. An alternative measuring apparatus may be used, provided that it has a resolution of 0,002 mm or finer.

5.4 Metallurgical microscope

A metallurgical microscope equipped with a photomicrographic capability or a measuring microscope shall be used to observe and measure chamfering of test piece edges.

6 Test piece dimension and preparation

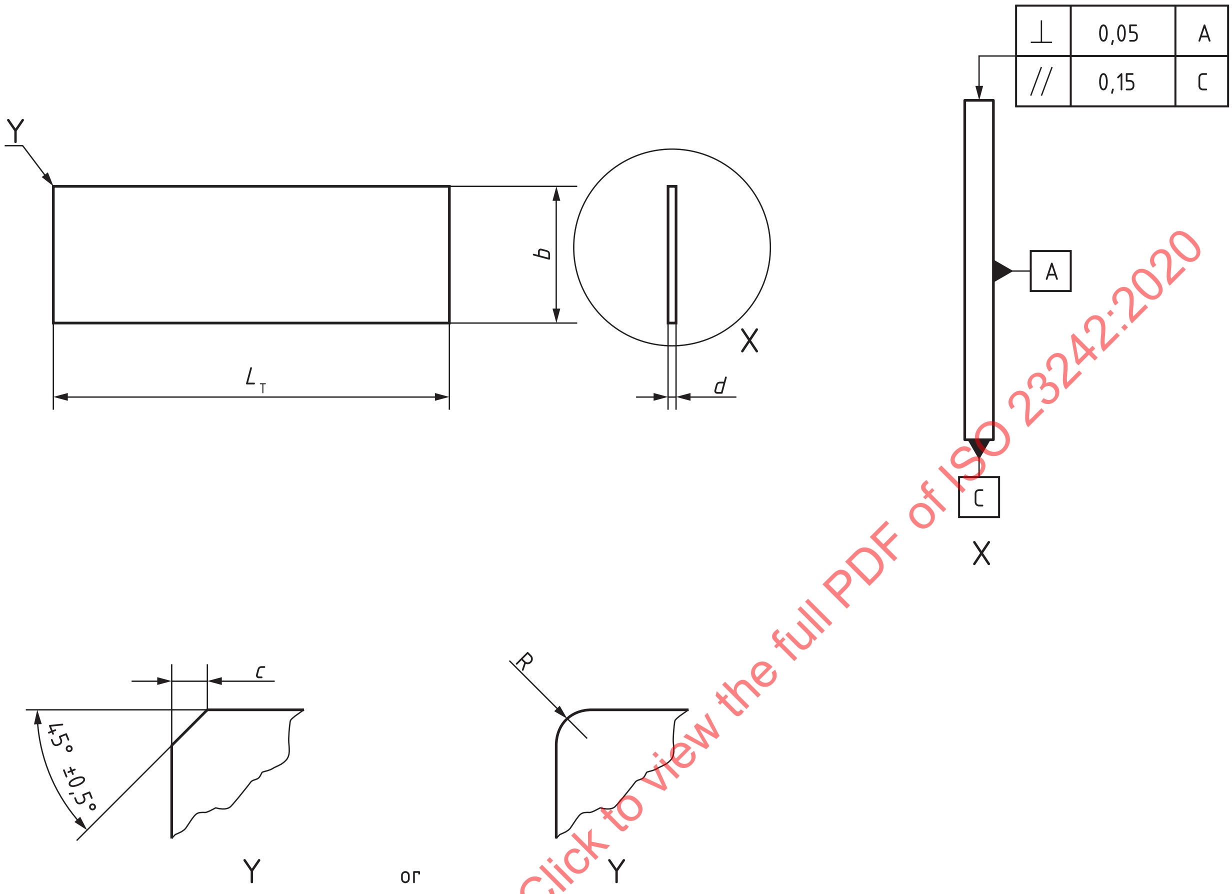
6.1 Shape and dimensions of test piece

6.1.1 Machined specimens

Test piece dimensions are shown in Figure 3. The parallelism tolerance on the opposite longitudinal side face $L \times d$ shall be 0,15 mm as defined in ISO 1101. The longitudinal side faces shall intersect with the large faces perpendicularly. The maximum variation in perpendicularity shall not exceed 0,05 mm. Variation of thickness $d_{max}-d_{min}$ shall be less than 10 % of the average thickness d_{avg}, which is determined from the thicknesses at three or more points.

6.1.2 As-fired specimens

Specimen dimensions may be altered as required, but deviations from the specifications in 6.1.1 and Figure 3 shall be stated in a test report.



Specimen type	Specimen thickness d mm	Total length L_T mm	Width b mm	Chamfering c mm	Rounded edge R mm
I	$0,5 \leq d \leq 1,0$	≥ 35	12 ± 0,2	0,05 to 0,13	0,05 to 0,13
II	$0,2 \leq d < 0,5$	≥ 20	12 ± 0,2	0,05 to 0,1	0,05 to 0,1

Figure 3 — Dimensions of specimen

6.2 Test piece preparation

6.2.1 General

This document allows several options for specimen preparation. All four long edges shall be chamfered or rounded as shown in Figure 3. It is highly recommended that the surface roughness be measured and reported, although a surface finish specification is not part of this document.

NOTE Machining damage can be introduced during surface preparation of test specimens (especially microcracks beneath the specimen surface) and can have an extensive effect on flexural strength. Machining flaws can be either a random interfering factor or an inherent part of the strength characteristics to be measured. Surface preparation can also create residual stresses. Final machining steps (including polishing) can negate machining damage introduced from prior, coarser machining steps.

6.2.2 As-fired

When the strength of a specimen with an as-fired surface is to be measured, the flexure specimen can be fabricated by sintering or some other process, and no machining is required. In this case, an edge chamfer or rounding is recommended.

As-fired specimens are particularly prone to twisting or warping. In these cases, a fully articulating fixture should be used in testing.

One surface of an as-fired specimen may be ground, polished or sand blasted to help reduce twisting or warping effects. The machined surface should be placed in contact with inner bearings (specimen compression side) during testing.

6.2.3 Customary machining procedure

In instances where a customary machining procedure has been developed that is completely satisfactory for a class of materials (i.e. it introduces minimal or no unwanted surface damage or residual stress), then this customary procedure is permitted. The test report shall include details of the procedure, especially the cutting device used (e.g. laser scribe, dicing), grinding wheel grits, wheel bonding (resin, metal, vitreous glass, other) and amount of material removed per pass. The long edges of the specimen shall be rounded or chamfered, as shown in Figure 3.

6.2.4 Component-matched procedure

The specimen shall have the same surface preparation as that given to a component. The test report shall include details of the procedure, especially the cutting device used (e.g. laser scribe, dicing), wheel bonding (resin, metal, vitreous, other) and the amount of material removed per pass. The long edges of the specimen shall be rounded or chamfered, as shown in Figure 3.

6.2.5 Handling of specimens

Specimens shall be handled with care in order to avoid introduction of damage after specimen preparation. Specimens shall be stored separately and not allowed to impact or scratch each other.

6.2.6 Number of test pieces

A minimum of 10 specimens shall be required for the purpose of estimating mean flexural strength. A minimum of 30 specimens shall be used if a statistical strength analysis (e.g. a Weibull analysis) is to be made.

7 Test procedure

7.1 Initial dimensions measurement for specimen type identification

Initial measurement of test piece thickness shall be done to identify the specimen type (I or II) as defined in Table 1 and then employ the appropriate bend test fixture (as described in 7.3). Measurement of test piece thickness and width shall be carried out using a micrometer or other device accurate to the nearest 0,002 mm. Dimensions shall be measured at one or more points along the specimen length but also at specific locations after fracture (as described in 7.11).

7.2 Specimen marking

Use a felt pen to gently mark the specimen to distinguish its compression surface from its tensile surface. Mark an inequality sign on the compression surface and the specimen number at both ends of the tensile surface. For fractographic analysis, the original test piece can be restored from the fractured fragments using this inequality sign. Carefully drawn felt-tip pen marks are preferable.

7.3 Bend test fixture

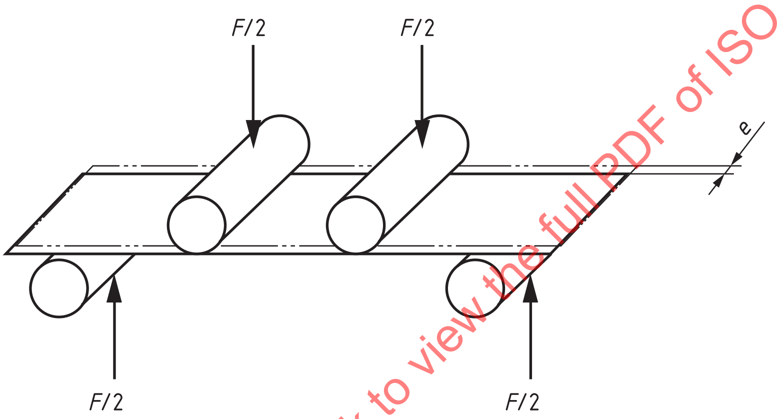
Employ an appropriate fixture in either a three-point or four-point configuration to test the specimens. A fully articulating fixture is preferred.

7.4 Fixture cleaning

Make sure that test fixtures are clean and free of any broken fragments left after former tests, and that bearings are free of deep abrasions or burrs and are easy to articulate and roll.

7.5 Setting test piece in bend fixture

Set each specimen in the test fixture with a wide face resting on the supporting bearings. Arrange the specimen carefully so that the specimen has a roughly identical degree of overhang beyond the two supporting rollers. Care should be taken to centre the specimen within 0,3 mm of the axis of load application (front to back), as illustrated in Figure 4. Use of appropriate setting guides for the specimen is preferable.



Key

e displacement, < 0,3 mm

F force

Figure 4 — Alignment of specimen under axis of load application

7.6 Pre-loading

Apply to the specimen a slight preload of no more than 10 % of the average strength so that all rollers contact the specimen evenly. Check that the bearings are in their correct starting positions.

7.7 Protection from fracture fragments

For safety, cover the test fixture with a protective screen such as a transparent polyethylene terephthalate (PET) film to trap fracture fragments.

7.8 Crosshead speed

Select a crosshead speed such that the time to fracture is within 5 s to 15 s, because some brittle ceramic materials are sensible to slow crack growth in specific test environments (humidity and temperature). If the operator chooses a low crosshead speed for the bending test, those mechanisms can strongly influence fracture strength measurement. Table 2 shows typical crosshead speed and strain rate for each test configuration. If the user can expect elastic modulus and fracture strength value of test material, the time to fracture can be roughly estimated by dividing the fracture strain by the strain rate shown in Table 2. Then the crosshead speed should be modified so that the time to failure is within 5 s to 15 s.

Table 2 — Typical crosshead speed and strain rate

Specimen type	Specimen thickness d mm	Bending mode	Typical crosshead speed mm/min	Strain rate $s^{-1}/10^{-4}$
I	$0{,}5 \leq d \leq 1{,}0$	3-point	2,34	1,3 to 2,6
I	$0{,}5 \leq d \leq 1{,}0$	4-point	2,34	1,2 to 2,3
II	$0{,}2 \leq d < 0{,}5$	3-point	1,17	1,0 to 2,6
II	$0{,}2 \leq d < 0{,}5$	4-point	1,17	0,9 to 2,3

7.9 Application of test force

Start loading at the specified rate and record the maximum force at fracture. Measure the maximum load with an accuracy of ± 1 % or better. Record the time to fracture.

7.10 Retrieval of broken fragments for successive measurements

After a sample is broken, collect fracture fragments and save only a few pieces. Tiny fragments or shards are usually of little importance because they do not contain the fracture origin. If possible, remove the loading member and all rollers from the main body of the fixture. Tiny fragments and debris shall be removed completely from both the main body of the fixture and rollers. It is recommended that the specimen be collected with tweezers after fracture or that the operator wears gloves in order to avoid contamination of the fracture surfaces for possible fractographic analysis using scanning electron microscopy or other such means.

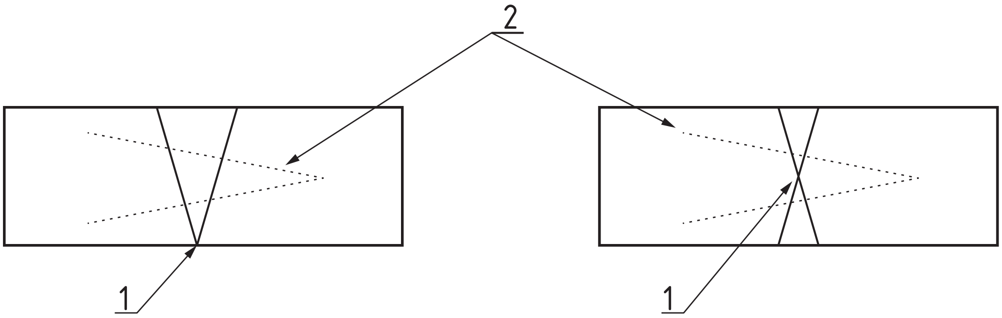
7.11 Measurement of test piece thickness and width

Specimen thickness and width shall be measured at three or more points. The recommended measurement points are a) near the fracture location and b) several millimetres from the fracture location (along the specimen length).

7.12 Fractography

Reassemble the original test piece from the fractured fragments using the inequality sign on the compression surface. Observe the approximate fracture origin to determine if fractures occurred at the edge of the test piece. The fracture origin is located at the centre of a radial crack pattern. Thus, the location of the fracture origin can be determined from the crack pattern. If the crack pattern has a “V” shape as in Figure 5, it is identified as an “edge failure.”

If many specimens fracture from the edge of the specimen, there can be a fixture misalignment. Testing shall be stopped until the problem is resolved.



a) Edge failure **b) Surface failure**

Key

1 fracture origin

2 inequality sign on the compression surface

Figure 5 — Identification of typical crack patterns using reassembled test specimens

8 Calculation

8.1 Three-point flexural strength

Calculate three-point flexural strength from the measured values for each specimen according to Formula (1).

$$\sigma_f = \frac{3FL}{2bd^2} \quad (1)$$

where

σ_f is the flexural strength, in MPa;

F is the fracture force, in N;

L is the length of the fixture lower span, in mm;

b is the average of specimen width at three or more points, in mm;

d is the average of specimen thickness at three or more points, in mm.

8.2 Four-point flexural strength

Calculate four-point flexural strength from the measured values for each specimen according to Formula (2).

$$\sigma_f = \frac{3Fa}{bd^2} \tag{2}$$

where

σ_f is the flexural strength, in MPa;

F is the fracture force, in N;

a is the length of the fixture moment arm, in mm;

b is the average of specimen width at three or more points, in mm;

d is the average of specimen thickness at three or more points, in mm.

8.3 Mean and standard deviation

Mean strength, $\overline{\sigma_f}$, and standard deviation, s, are given by Formulae (3) and (4):

$$\overline{\sigma_f} = \frac{\sum_1^n \sigma_{f,i}}{n} \tag{3}$$

$$s = \left[\frac{\sum_1^n \left(\sigma_{f,i} - \overline{\sigma_f} \right)^2}{n-1} \right]^{1/2} \tag{4}$$

where

$\sigma_{f,i}$ is the strength of the i th specimen;

n is the total number of specimens.

If the chamfer sizes are larger than those specified in 6.1.1 and Figure 3, then the flexural strength should be corrected as specified in Annex A.

9 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 23242:2020;

b) test configuration (four- or three-point flexure), fixture size, fixture articulation (semi-articulating or fully articulating) and a statement affirming that bearings were free to roll;

c) number of specimens (n) tested;

d) all relevant material data, including batch, billet or component identification number (the date the material was manufactured should be reported, if available);

e) specimen preparation procedures, including all details of machining preparation;

f) heat treatments or exposures, if any;

g) flexural test environment details, including humidity and temperature;

h) crosshead speed, in mm/min, and approximate average time-to-fracture, in seconds;

i) for each specimen tested, the flexural strength ($\sigma_{f, i}$) to three significant figures (e.g. 537 MPa);

j) mean strength ($\overline{\sigma_f}$) and the standard deviation (s);

k) a statement reporting whether chamfers are within specification; if not, then a statement clarifying whether flexural strength has been corrected for chamfer size;

l) any deviation(s) from the procedures described in this test method, and the reason for the deviation(s);

m) name of testing laboratory, date of test, name of person conducting tests and name of testing machine.

10 Weibull size scaling

Different mean strength can be obtained when the different sample dimensions and fixture sizes are employed. Weibull size scaling can facilitate comparison of results. See ISO 14704:2016, Annex E.

