INTERNATIONAL STANDARD

ISO 14704

Third edition
2016-04-15

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for flexural strength of monolithic ceramics at room temperature

Céramiques techniques — Méthode d’essai de résistance en flexion des céramiques monolithiques à température ambiante

Reference number
ISO 14704:2016(E)

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 206, *Fine ceramics*.

This third edition cancels and replaces the second edition (ISO 14704:2008), which has been technically revised.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for flexural strength of monolithic ceramics at room temperature

1 Scope

This International Standard specifies a test method for determining the flexural strength of monolithic fine ceramics, and whisker- or particulate-reinforced ceramic composites, at room temperature and applies to materials with grain size less than 200 µm. This test method may be used for materials development, quality control, characterization and design data-generation purposes.

NOTE Since fracture is due to tensile stress, flexural strength data can be used to calculate a uniaxial tensile strength considering the effect of the tested volume and Weibull-statistics. So, flexural strength is often used in substitute for uniaxial tensile strength.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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*

3 Terms and definitions

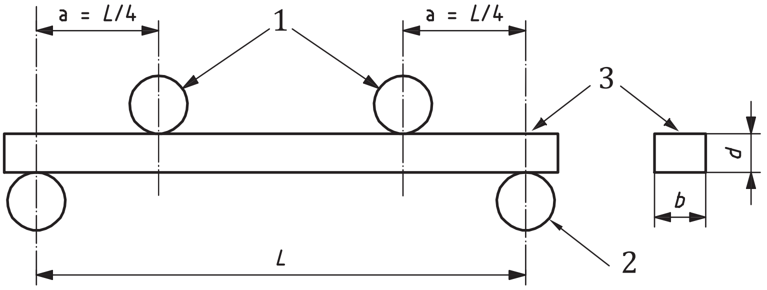
For the purposes of this document, the following terms and definitions apply.

3.1
flexural strength
maximum nominal stress at fracture of a specified elastic beam loaded in bending

3.2
four-point flexure
configuration of flexural strength testing where a test piece is loaded equally by two bearings symmetrically located between two support bearings

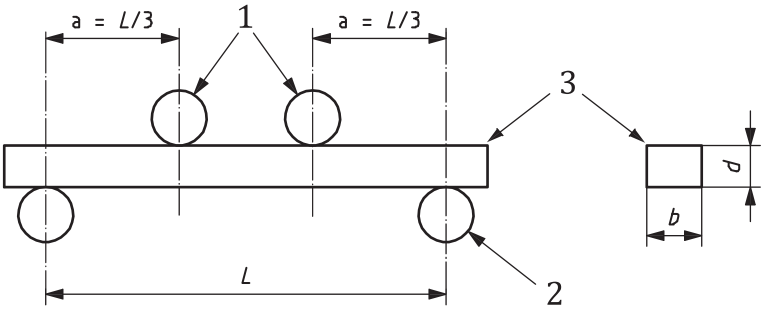
Note 1 to entry: See Figure 1, a) and b).

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



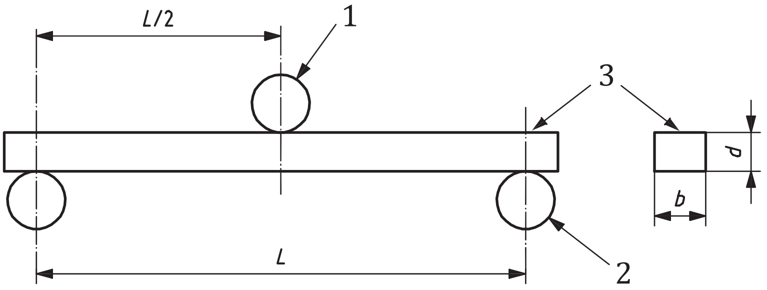
L = 40 mm ± 0,1 mm

a) Four-point-1/4 point flexure



L = 30 mm ± 0,1 mm

b) Four-point-1/3 point flexure



L = 30 mm ± 0,1 mm or 40 mm ± 0,1 mm

c) Three-point flexure

Key

1 loading bearings

2 support bearing

3 test piece

Figure 1 — Flexural test configurations

3.3
four-point-1/4 point flexure

specific configuration of four-point flexural strength testing where the inner bearings are situated one-quarter of the support span away from the two outer bearings

Note 1 to entry: See Figure 1 a).

3.4

four-point-1/3 point flexure

specific configuration of four-point flexural strength testing where the inner bearings are situated one-third of the support span away from the two outer bearings

Note 1 to entry: See Figure 1 b).

3.5

semi-articulating fixture

test fixture designed to apply uniform and even loading to test pieces that have flat and parallel surfaces

3.6

fully articulating fixture

test fixture designed to apply uniform and even loading to test pieces that may have uneven, non-parallel or twisted surfaces

3.7

three-point flexure

configuration of flexural strength testing where a test piece is loaded at a location midway between two support bearings

Note 1 to entry: See Figure 1 c).

Note 2 to entry: *Four-point flexure* (3.2) is usually preferred, since a large amount of material is exposed to the maximum stress (see Annex A for more information).

4 Principle

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

5 Apparatus

5.1 Testing machine

The testing machine shall be capable of applying a force to the loading roller (three-point flexure) or equally to the loading rollers (four-point flexure) in order to stress the test piece. The machine shall be capable of applying the force at a constant loading or displacement rate. The test machine shall be equipped for recording the peak load applied to the test piece. The accuracy of the test machine shall be in accordance with ISO 7500-1, Class 1, with an accuracy of 1 % of indicated load at fracture.

5.2 Test fixture

5.2.1 General

Three- or four-point flexure configurations shall be used, as illustrated in Figure 1. The four-point-1/4 point configuration is recommended. The fixture shall have bearings that are free to roll, as described in 5.2.2, in order to eliminate frictional constraints when the test piece 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 test piece. The articulation is designed so that parts of the fixture can rotate, as shown in Figure B.1, to ensure even loading on the left and right bearings. An articulation is also needed to ensure that all the bearings evenly contact the test piece surfaces and apply uniform load. Semi-articulated fixtures have some articulating or tilting capabilities and may be used with test pieces that have flat and parallel surfaces, such as on as-machined test pieces. A semi-articulating fixture has pairs of upper and lower bearings that articulate to match the test piece surfaces, as shown in Figures B.2 and B.3. Fully articulated fixtures have more moving parts and are necessary for test pieces that do not have flat and parallel surfaces. They allow independent articulation of the bearings. Fully articulated fixtures often are necessary for as-fired, heat-treated or oxidized test pieces, since uneven loading can cause twisting and severe errors. A fully articulating fixture may also be used with machined test pieces.

5.2.2 Bearings

Test pieces shall be loaded and supported by bearings. The bearings may be cylindrical rollers or cylindrical bearings. The bearings shall be made of a steel which has a hardness of no less than HRC 40 for test piece strengths up to 1 400 MPa, or no less than HRC 46 for test piece strengths up to 2 000 MPa. Alternatively, the bearing may be made of a ceramic or hardmetal 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 12 mm. The bearing diameter shall be approximately 1,5 times the test piece thickness (d). Diameters between 4,5 mm and 5 mm are recommended. 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. In four-point flexure, the two inner bearings shall be free to roll inwards, and the two outer bearings shall be free to roll outwards. In three-point flexure, the two outer bearings shall be free to roll outwards, and the inner (middle) bearing shall not roll.

NOTE 1 Friction can cause errors in the stress calculations. The rolling can be accomplished by several designs. The bearing can be mounted in roller bearing or cylindrical bearing assemblies. It is also acceptable, and simpler, for the bearings to be free to roll on the fixture surface, as shown in Figure 2.

The bearing diameter is specified on the basis of competing requirements. The bearings should not be so large as to cause excessive change in the moment arm as a test piece deflects, as this can create errors from contact-point tangency shift. On the other hand, the bearings should not be so small as to create excessive wedging stresses in the test piece or create contact stresses that damage the fixture.

NOTE 2 The bearing hardness and stiffness requirements and guidelines are intended to ensure that test pieces with strengths up to 1 400 MPa (or 2 000 MPa), and elastic moduli as high as 500 GPa, can be tested without damaging the fixture. Higher-strength or stiffer ceramic test pieces can require harder bearings. For example, if the bearing elastic modulus is greater than 500 GPa, then it is advisable to lengthen the bearings and the fixture support width to more than 12 mm to distribute the forces over a longer bearing length.

5.2.3 Four-point fixture: semi-articulating

Figure B.2 a) shows the actions of the bearings in this fixture. The two inner bearings shall be parallel to each other to within 0,015 mm over their length (≥ 12 mm in accordance with 5.2.2). The two outer bearings shall be parallel to each other to within 0,015 mm over their length. Either the two inner or the two outer bearings shall be capable of articulating (tilting) together as a pair to match the test piece surface. All four bearings shall rest uniformly and evenly across the test piece surface. The fixture shall apply equal load to all four bearings. All four bearings shall be free to roll.

5.2.4 Four-point fixture: fully articulating

Figure B.2 b) shows the actions of the bearings in this fixture. One bearing need not articulate (tilt). The other three bearings shall articulate (tilt) independently to follow the test piece surface. All four bearings shall rest uniformly and evenly across the test piece surface. The fixture shall apply equal load to all four bearings. All four bearings shall be free to roll.

5.2.5 Three-point fixture: semi-articulating

Figure B.3 a) shows the actions of the bearings in this fixture. The two outer bearings shall be parallel to each other to within 0,015 mm over their length (≥ 12 mm in accordance with 5.2.2). The two outer bearings shall articulate together to follow the test piece surface, or the middle bearing shall articulate to follow the test piece surface. All three bearings shall rest uniformly and evenly across the test piece surface. The fixture shall be designed to apply equal load to the two outer bearings. The two support (outer) bearings shall be free to roll outwards. The middle bearing shall be fixed and not free to roll.

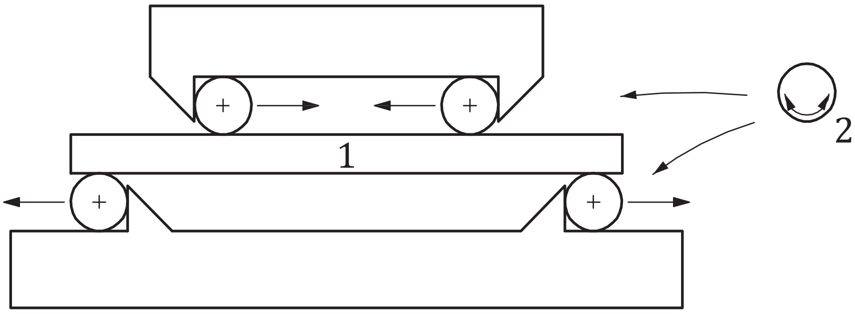
5.2.6 Three-point fixture: fully articulating

Figure B.3 b) and c) show the actions of the bearings in this fixture. Any two of the bearings shall be capable of articulating (tilting) independently to rest uniformly and evenly across the test piece surface. The fixture shall be designed to apply equal load to the two outer bearings. The two support (outer) bearings shall be free to roll outwards. The middle bearing shall not roll.

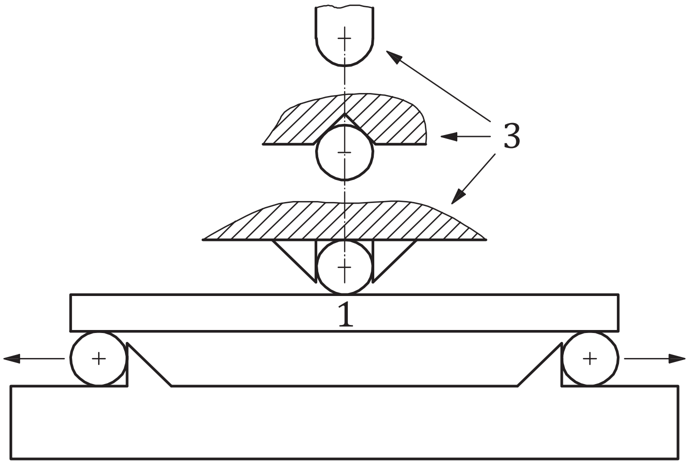
5.2.7 Positioning of bearings

The bearings shall be positioned so that the spans are accurate to within ±0,1 mm. The middle bearing for the three-point fixture shall be positioned midway between the outer bearings to within ±0,1 mm. The inner bearings for the four-point fixture shall be centered 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 traveling microscope or other suitable device. The spans can also be verified by measurement of the distances between bearing stops and adding (outer span) or subtracting (inner span) the radii of the bearing cylinders.



a) Four-point flexure



b) Three-point flexure

Key

1 test piece

2 alternative rolling bearings

3 alternative loading bearing arrangements

NOTE 1 For a), the four bearings shall be free to roll.

NOTE 2 For b), the two outer bearings are free to roll outwards, but the middle bearing shall be non-rolling.

Figure 2 — Schematic representation of fixtures showing the rolling action of the bearing

5.2.8 Fixture material

The fixture which supports and aligns the bearings shall be sufficiently hard, so that the bearings do not permanently deform the fixture.

NOTE Line-contact loadings can deform the fixture. The hardness of the fixture will depend upon the design of the fixture. If the bearings are at least 12 mm wide and the fixture is 12 mm wide or more, then a fixture made of steel with an HRC of 25 or greater will be adequate.

5.3 Micrometer

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

6 Test pieces

6.1 Test piece size

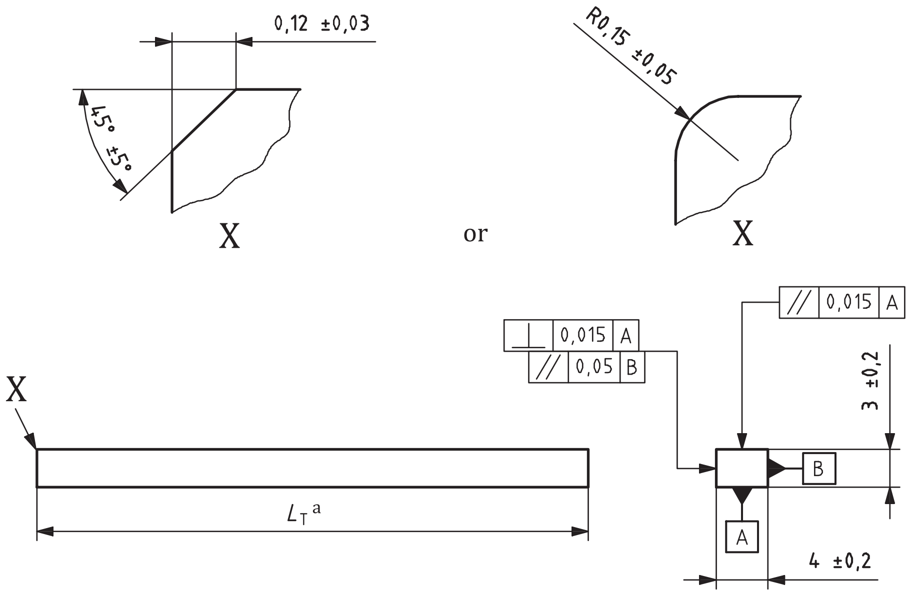
6.1.1 Machined test pieces

Test piece dimensions are shown in Figure 3. Cross-sectional tolerances shall be ±0,2 mm. The parallelism tolerance on opposite longitudinal faces is 0,015 mm.

6.1.2 As-fired or heat-treated test pieces

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

Dimensions in millimetres



Key

a $L_T \geq 35$ mm for 30 mm test fixtures and $L_T \geq 45$ mm for 40 mm test fixtures.

Figure 3 — Standard test piece

6.2 Test piece preparation

6.2.1 General

This International Standard allows several options for test piece preparation. In all cases, the end faces of the test piece do not need special preparation or finishing. A minimum of two long edges on one 4 mm wide face shall be chamfered or rounded, as shown in Figure 3. It is highly recommended that all four long edges be chamfered or rounded. The test piece surface condition (as-fired, ground, polished etc.) shall be reported.

Although a surface finish specification is not part of this International Standard, it is highly recommended that the surface roughness be measured and reported.

NOTE Surface preparation of test pieces can introduce machining flaws (especially microcracks beneath the test piece surface) which can have a pronounced effect on flexural strength. Machining damage can either be 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 or cannot negate machining damage introduced from prior, coarser machining steps.

6.2.2 As-fired

The flexure test piece is fabricated by sintering or some other process, such that no machining is required. In this case, the purpose is to measure the strength of the test piece with an as-fired surface. An edge chamfer or rounding is recommended and can be made before sintering.

As-fired test pieces are especially prone to twist or warpage. They may not meet the parallelism requirements given in 6.1.1, in which case a fully articulating fixture should be used in testing.

One surface of an as-fired part may be machined to help minimize twisting or warpage effects. The machined surface should be placed in contact with the inner bearings (test piece 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 wheel grits, wheel bonding (resin, metal, vitreous glass, other) and the material removed per pass. The long edges of the test piece shall be rounded or chamfered, as shown in Figure 3.

6.2.4 Component-matched procedure

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

6.2.5 Basic machining procedure

If the procedures in 6.2.2 to 6.2.4 are not applicable, then the following procedure may be used.

NOTE The procedure specified below is a general-duty, conservative practice. It is intended to minimize machining damage or residual stresses in a broad range of ceramics. Faster or more aggressive removal rates can be suitable for some materials. Alternatively, some very brittle ceramics can require a more conservative preparation.

6.2.5.1 Test pieces shall be ground in the longitudinal direction, as shown in Figure 4.

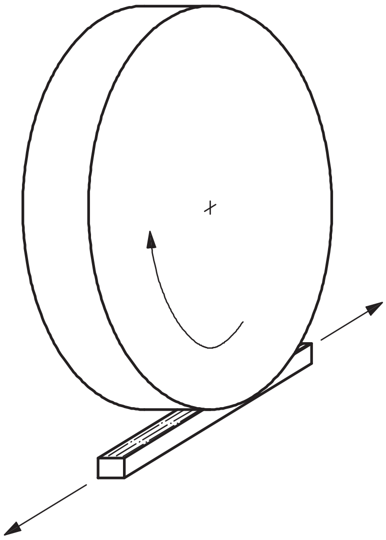


Figure 4 — Surface grinding parallel to the test piece longitudinal axis

6.2.5.2 All grinding shall be done with an ample supply of filtered coolant, in order to keep the work piece and wheel flooded and particles flushed. Grinding shall be in at least two stages, ranging from coarse to fine rates of material removal.

6.2.5.3 Coarse grinding shall be carried out using a diamond wheel rounded to within 0,03 mm and of grit size not exceeding 120 mesh (D 126), using a depth of cut not exceeding 0,03 mm per pass. Alternatively, a creep-feed grinding process may be used for the coarse grinding step.

6.2.5.4 Finishing machining shall be carried out using a diamond wheel of grit size between 320 mesh and 800 mesh (e.g. D 46 or finer), using a depth of cut not exceeding 0,002 mm per pass. Final finishing shall remove no less than 0,06 mm of material per face. Approximately, equal stock shall be removed from opposite faces.

6.2.5.5 The long edges shall be uniformly chamfered at 45° to a size of 0,12 mm ± 0,03 mm, as shown in Figure 3. Alternatively, they can be rounded to a radius of 0,15 mm ± 0,05 mm. Edge chamfering or rounding shall be comparable to that applied to the test piece surfaces in the fine-finishing step. The direction of machining shall be parallel to the test piece's long axis.

If, for some reason, the chamfers are larger than the specified size range (e.g. for the removal of very large chips), then the stresses should be corrected for the reduced second moment of inertia of the test piece cross-section. Annex D may be consulted for this correction.

6.2.5.6 The final dimensions of the test piece shall be in accordance with 6.1.1 and Figure 3.

6.2.6 Parallelism, orthogonality and chamfer sizes

Ensure that the parallelism, orthogonality and chamfer sizes of the test pieces are checked. If the test pieces have been prepared by an established procedure with a demonstrated reliability, then inspect only a few (three to five per batch) test pieces to verify conformance. The basis for acceptance/rejection of parallelism shall be by measurements made across the thickness and across the width at each end of the intended support span and in the center. A flat-faced hand micrometer, dial indicator/comparator stand or digital indicator stand may be used. The acceptability for orthogonality shall be based on the use of an engineering shadowgraph, optical comparator or optical microscope. The basis for chamfers shall be based on microscope examination and measurement.

6.2.7 Handling of test pieces

The test pieces shall be handled with care in order to avoid the introduction of damage after test piece preparation. Test pieces shall be stored separately and not allowed to impact or scratch each other.

6.2.8 Number of test pieces

A minimum of 10 test pieces shall be required for the purpose of estimating the mean flexural strength. A minimum of 30 test pieces shall be used if a statistical strength analysis (for example, a Weibull analysis) is to be made.

NOTE The use of 30 test pieces will help obtain good confidence limits for the strength distribution parameters such as a Weibull modulus. The 30 test pieces will also help detect multiple flaw populations if they exist.

7 Procedure

7.1 Measure the test piece width, b, and thickness, d, with a resolution of 0,002 mm. The test piece size may be measured either before or after the test. If the test piece is measured before the test or if there is excessive fragmentation, measure the test piece dimensions as close to the midpoint (along the test piece length) as possible; otherwise, measure the test piece dimensions at or near the fracture location after the test. Care shall be taken to not introduce surface damage when using the micrometer.

7.2 Test the test pieces on the appropriate fixture in either the three- or four-point configuration. The four-point configuration is preferred. A fully articulating fixture shall be used if the test piece parallelism requirements cannot be met.

7.3 Ensure that the test fixtures are clean and free of any fracture debris from previous tests, that the bearings are free of burrs or deep scratches and that the bearings are free to roll and articulate.

7.4 Place each test piece in the test fixture with a 4 mm wide face resting on the bearings. If the test piece has only two edges chamfered or rounded, place the test piece so that these chamfers are on the tension side. Avoid damaging the test piece. Align the test piece carefully. The test piece should have an approximately equal amount of overhang beyond the two outer bearings. Centre the test piece carefully within 0,1 mm of the axis of load application (front to back), as illustrated in Figure 5. Positioning stops for the test piece are strongly recommended.

This is especially important with fully articulating fixtures which may cause the test piece to shift during articulation. The fixture design should not allow excessive shifts.

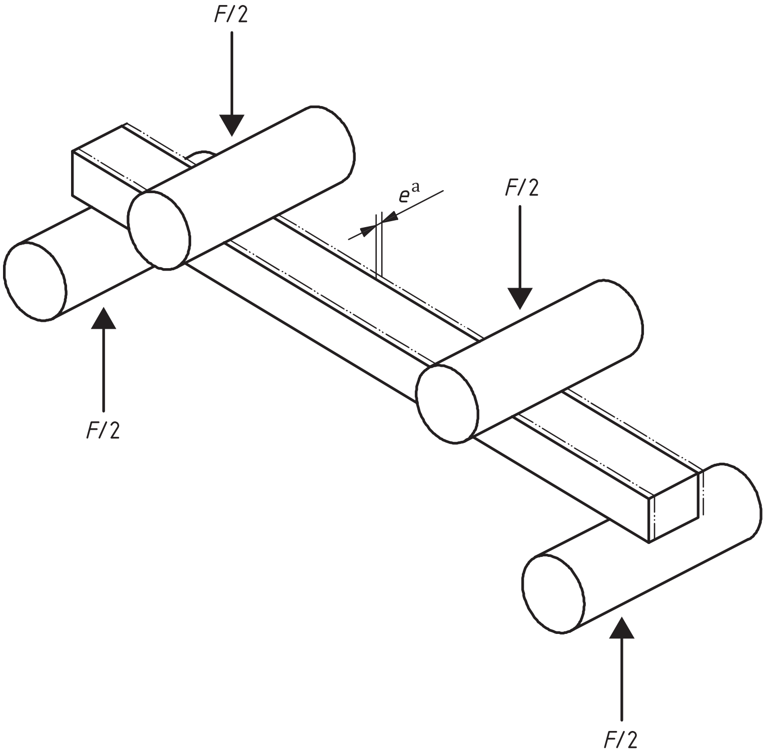
7.5 Apply to the test piece a slight preload of no more than 10 % of the mean strength. If possible, inspect the lines of contact of all the bearings and the test piece to ensure that there is an even line loading. If the loading is not even, then unload the test piece and adjust the fixtures as required to obtain even loading. Inspect the bearings to ensure that they are in their correct starting positions.

7.6 Gently mark the test piece to identify the approximate locations of the two inner loading bearings (four point) or the middle bearing (three point). Also, mark the test piece so that the compression surface can be distinguished from the tensile surface. Carefully drawn pencil or felt-tip pen marks are suitable.

7.7 Place cotton, tissue, foam or another material around or near the test piece to prevent the test piece fragments from flying around the fixture after fracture. These materials shall not interfere with the load application or inhibit fixture articulation or bearing rolling motions.

NOTE 1 This will prevent unnecessary secondary fractures and will help preserve the primary fracture pieces for subsequent fractographic analysis when needed.

NOTE 2 For safety reasons, place a protective screen around the test fixture to trap the fracture fragments.



Key

[a] $e < 0,1$ mm.

Figure 5 — Alignment of the test piece under the axis of load application

7.8 Select a rate of load application such that the time to fracture is within 5 s to 15 s.

NOTE 1 Selection of crosshead displacement rate may have to be determined by experiment, depending on the elastic compliance of the test machine, the stiffness of the test fixture and the elastic properties of the test piece. A crosshead displacement rate of typically 0,5 mm/min is a convenient starting point for most testing machines in cases where the expected strength of the material is 200 MPa to 400 MPa. For materials which are much weaker or much stronger than this, the displacement rate may have to be respectively decreased or increased by an appropriate factor.

NOTE 2 The strength of fine ceramics may be markedly affected by the testing rate. The short time to fracture required by this method is intended as a compromise between the elimination of load-rate effects and the ability of test machines and load recording equipment to provide an accurate measure of peak fracture load.

NOTE 3 The effects of time-dependent phenomena, such as stress or slow crack growth corrosion (from water vapor in laboratory ambient air) on the measured flexural strength can be important for some sensitive materials, even for the relatively short times in the test. To eliminate or minimize the environmental sensitivity, surround the test fixture with an environmental chamber or envelope (for example, a clear polyethylene bag) and flush with dry nitrogen or argon from a gas cylinder before the test. Run the test with inflowing dry nitrogen or argon gas. Alternatively, coat the tensile face of the test pieces with paraffin oil (preferably heat treated at approximately 170 °C for 1 h to 2 h) or silicone oil and then test the test piece in laboratory ambient conditions. The paraffin oil may interfere with subsequent fractographic analysis, however. Alternatively, the tests may be done in a vacuum (~ 10^{-3} Pa or less).

7.9 Apply the test force at the chosen rate and record the peak load at fracture. Record the time to fracture.

7.10 Retrieve the fracture fragments for later examination.

Only a few pieces need be saved. Tiny fragments or tiny shards are often inconsequential since they do not contain the fracture origin. With experience, it is usually not difficult to determine which pieces are important and should be retained. Annex C may be consulted for further guidance. It is recommended that the test piece be retrieved with tweezers after fracture, or the operator may wear gloves, in order to avoid contamination of the fracture surfaces for possible fractographic analysis.

7.11 Observe the approximate fracture-initiation location with the primary purpose of observing if fractures occurred within the inner span in four-point flexure. Fracture of test pieces may occasionally occur outside the inner span or at an inner loading bearing. These test pieces shall be included in the data set. See Annex C for more guidance on the interpretation of fracture patterns in flexural test pieces.

NOTE 1 This is a normal consequence of the scatter in fracture-origin sizes and locations. Fractures from origins outside the inner span are more likely to occur with materials with high strength scatter, i.e. a low Weibull modulus.

If many test pieces fracture outside the inner span, or if many fractures begin directly under the inner loading bearings in four-point flexure, there may be a fixture misalignment. Testing shall be stopped until the problem is remedied.

NOTE 2 Multiple fractures are common in high-strength ceramics. In many instances, a secondary fracture will occur directly under an inner bearing. This is common and the strength result is probably completely satisfactory. Guidance on the interpretation of fracture origins and patterns is included in Annex C.

7.12 Measure and record the relative humidity and ambient temperature during the test sequence.

8 Calculation

8.1 The standard formula for the flexural strength in four-point flexure is

$$\sigma_{\mathrm{f}} = \frac{3Fa}{bd^2} \tag{1}$$

where

σ_{f} is the flexural strength, in MPa;

F is the fracture load, in N;

a is the length of the fixture moment arm (10 mm);

b is the test piece width, in mm;

d is the test piece thickness, parallel to the direction of test force, in mm.

NOTE 1 a is 10 mm for both fixtures specified by this International Standard; $a = L/4$ for 1/4-point, four-point flexure and $a = L/3$ for 1/3-point, four-point flexure (see Figure 1).

NOTE 2 Formula 1 gives the nominal flexural strength of a test piece. See the last paragraph of 8.2.

8.2 The standard formula for the flexural strength in three-point flexure is

$$\sigma_{\mathrm{f}} = \frac{3FL}{2bd^2} \tag{2}$$

where

σ_f is the flexural strength, in MPa;

F is the fracture load, in N;

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

b is the test piece width, in mm;

d is the test piece thickness, parallel to the direction of test force, in mm.

NOTE The three-point fixture outer span is either 40 mm or 30 mm, as specified in this International Standard.

Formulae 1 and 2 are the customary and correct formulae for reporting the nominal flexural strength of a test piece. They give the maximum stress occurring in the test piece at the instant of fracture. The formulae do not necessarily give the stress that was acting directly upon the flaw that caused fracture. In some instances, for example, fracture mirror or fracture toughness calculations, the fracture stress should be corrected for subsurface origins, breaks outside the inner span in four-point flexure or fractures not directly beneath the middle bearing in three-point flexure.

8.3 If the chamfer sizes are larger than those specified in 6.2.6 and Figure 3, then the flexural strengths should be corrected, as described in Annex D.

8.4 The mean strength, $\bar{\sigma}_f$, and the standard deviation, s, are given by

$$\bar{\sigma}_f = \frac{\sum_I^n \sigma_{f,i}}{n} \qquad (3)$$

$$s = \left[\frac{\sum_I^n (\sigma_{f,i} - \bar{\sigma}_f)^2}{n-1} \right]^{1/2} \qquad (4)$$

where

$\sigma_{f,i}$ is the strength of the ith test piece;

n is the total number of test pieces.

9 Test report

The test report shall be in accordance with the reporting provisions of ISO/IEC 17025 and shall include at least the following:

a) a reference to this International Standard, i.e. ISO 14704;

b) the test configuration (four- or three-point flexure), the fixture size, a statement on whether the fixture was semi-articulating or fully articulating and a statement that confirms that the bearings were free to roll;

c) the number of test pieces (n) tested;

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

e) test piece preparation procedures, including all details of machining preparation, if known;

f) heat treatments or exposures, if any;

g) flexural test environment, including humidity and temperature;

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

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

j) the mean strength, σ_f, and the standard deviation, s;

   The following notation shall be used to report the mean strengths:

   1) $\sigma_{(N,L)}$ to denote strengths measured in (N = 4 or 3)-point flexure, and (L = 40 mm or 30 mm) fixture outer-span size;

   EXAMPLES

   — $\sigma_{(4,40)}$ = 537 MPa denotes a mean flexural strength of 537 MPa when measured in four-point flexure with 40 mm span fixtures;

   — $\sigma_{(3,30)}$ = 580 MPa denotes a mean flexural strength of 580 MPa when measured in three-point flexure with 30 mm span fixtures;

   It is also recommended that the relative humidity or test environment be reported as follows:

   2) $\sigma_{(N,L)}$ = XXX [relative humidity (RH) % or environment] to denote strengths measured in an atmosphere with RH % or other environmental conditions;

   EXAMPLES

   — $\sigma_{(4,40)}$ = 537 MPa (45 %) denotes a mean flexural strength of 537 MPa, when measured in four-point flexure with 40 mm span fixtures in laboratory ambient conditions at 45 % relative humidity;

   — $\sigma_{(3,30)}$ = 580 MPa (dry N_2) denotes a mean flexural strength of 580 MPa, when measured in three-point flexure with 30 mm span fixtures in a dry-nitrogen-gas environment;

   — $\sigma_{(3,30)}$ = 580 MPa (paraffin oil coated) denotes a mean flexural strength of 580 MPa, when measured in three-point flexure with 30 mm span fixtures with paraffin-oil-coated test pieces;

k) a statement reporting whether the chamfers are within specifications; if they are not, then include a statement whether flexural strength has been corrected for the 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 the tests, name of testing machine.

10 Strength scaling factors

The different test pieces and fixture sizes permitted by this International Standard may produce different mean strengths. Annex E includes Weibull strength scaling factors which may facilitate comparison of results.

Annex A
(informative)

General information

The flexural strength of a ceramic depends on the inherent resistance to fracture and the presence of fracture origins in the ceramic; variations in these cause a natural scatter in test results for a sample of test pieces. Fractographic analysis of fracture surfaces, although beyond the scope of this International Standard, is highly recommended for all purposes, especially if the data will be used for design.

Flexural strength may also be influenced by many parameters associated with the test procedure. These include the loading rate, test environment, test piece size, test fixture details and test piece preparation. Surface preparation is especially important, since the maximum stress at fracture is on the test piece surface. With proper care and good machining practice, it is possible to obtain fractures from the material's natural flaws; otherwise, the test may only measure machining damage. This International Standard allows several options for the machining of test pieces and includes a general procedure ("Basic" machining procedure; see 6.2.5) which is satisfactory for many (but certainly not all) ceramics. The general procedure uses progressively finer longitudinal grinding steps that are designed to minimize subsurface microcracking. Longitudinal grinding aligns the most severe microcracks parallel to the test piece tension-stress axis. This allows a greater opportunity of measuring the "potential strength" of the material, as controlled by the material's natural flaws. In contrast, transverse grinding aligns the severest machining microcracks perpendicular to the tension-stress axis and the test piece is more likely to fracture from the machining microcracks. Transverse-ground test pieces in many instances may provide a more "practical strength" which is relevant to machined ceramic components, whereby, it may not be possible to favorably align the machining direction.

The specifications given in this International Standard were chosen to provide a balance between controlling experimental error, maintaining practicality and efficiency of testing. If the procedures in this International Standard are followed, it is estimated that the error in flexural strength for one test piece is less than 2 %.

Two fixtures and corresponding test piece sizes have been adopted in this International Standard. The larger fixture (20 mm × 40 mm) had previously been standardized in the United States and Europe. The shorter fixture (10 mm × 30 mm) had previously been standardized in Japan, Korea and the People's Republic of China. The larger fixture-test piece set will expose more material to the full stress and, in general, produce a slightly lower strength than the shorter fixture-test piece set. This is normal for ceramics wherein fracture origins of different size or severity are distributed through the volume or surface. Weibull statistics often can correlate the strengths quite efficiently, for example, for a material with a single volume-distributed fracture-origin type and a Weibull modulus of 10, the larger four-point test piece should have a strength 6 % lower than the strength for the smaller test piece.

The three-point test configuration exposes only a very small portion of the test piece to the maximum stress. Therefore, strengths are likely to be much higher than four-point flexural strength. Three-point flexure has some advantages. It uses simpler test fixtures, it is easier to adapt to high temperature and fracture toughness testing and it is sometimes helpful in Weibull statistical studies. Four-point flexure is preferred and recommended for most characterization work.

For additional information on flexural testing in general and for design, consult Reference [4]. For additional information on experimental errors in flexural testing of ceramics including correction factors for oversized chamfers, consult Reference [2].

Annex B
(normative)

Test fixtures

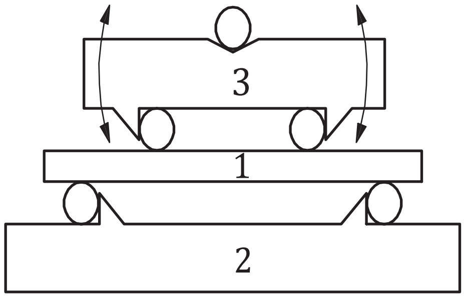
The fixtures shall articulate (tilt) to ensure even loading from side to side, as shown in Figure B.1. Additional articulation is necessary, as described below, to ensure that the bearings make even contact on the test piece top and bottom surfaces.

Semi-articulating fixtures are designed to apply uniform and even loading to test pieces that have flat and parallel surfaces. The top or bottom pair of bearings shall articulate to match the test piece surface, as shown in Figures B.2 a) and B.3 a).

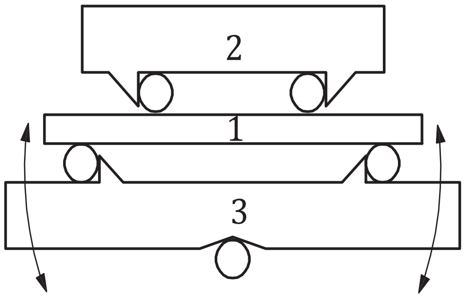
Fully articulating fixtures shall be used for test pieces that do not meet the test piece parallelism requirements of 6.1.1 and Figure 3. The bearings articulate independently to match the test piece surface, as shown in Figures B.2 b) and B.3 b). Fully articulating fixtures may also be used with flat and parallel test pieces.

Figures B.1 to B.3 illustrate fully and semi-articulating fixture actions. Figures B.4 and B.5 show some examples of designs. Other designs may be acceptable.

All fixtures shall have bearings that are free to roll, with the exception that the middle bearing in three-point flexure shall be non-rolling.



a) Upper pair of bearings which articulate



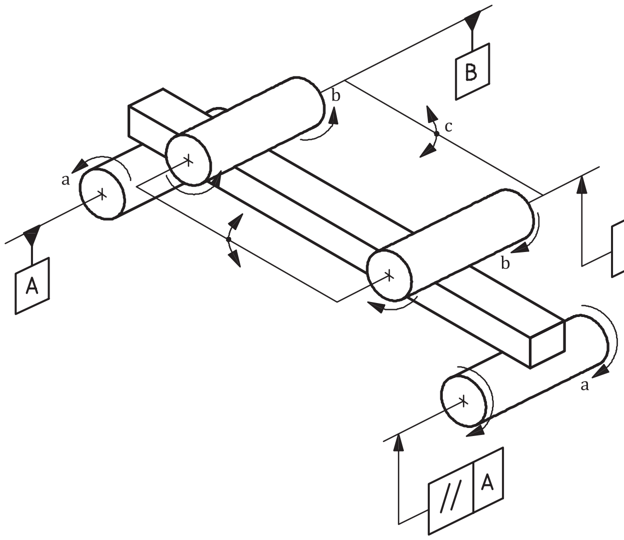
b) Lower pair of bearings which articulate

Key

1	test piece
2	fixed
3	articulating

NOTE Both arrangements are acceptable.

Figure B.1 — Four-point flexure fixture with top or bottom articulation to ensure even loading from side to side



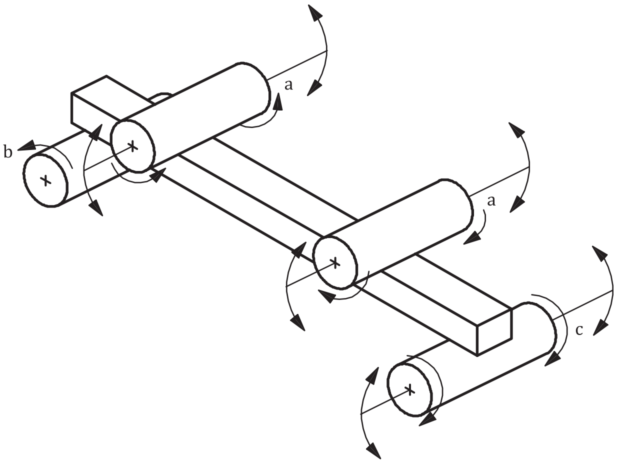
a) Semi-articulating

Key

a The outer bearings are parallel and free to roll outwards.

b The inner bearings are parallel and free to roll inwards.

c The inner bearings articulate together as a pair to follow the test piece top surface. (Alternatively, the two bottom bearings can articulate to follow the bottom surface).



b) Fully articulating

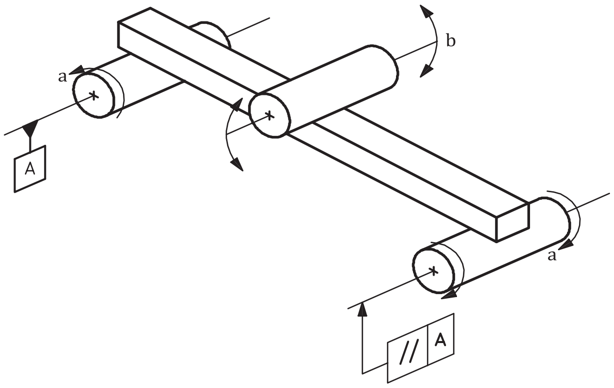
Key

a The inner bearings articulate and are free to roll inwards, and they can articulate independently to follow the test piece top surface.

b Not articulating (fixed) but free to roll.

c Articulating and free to roll outwards.

Figure B.2 — Four-point flexure fixture

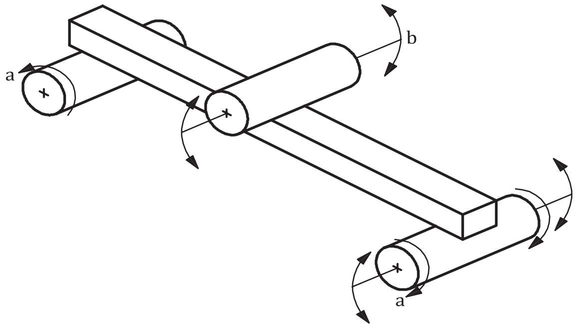


a) Semi-articulating

Key

a The two support bearings are parallel to each other and free to roll outwards.

b The middle bearing does not roll, but it can articulate to follow the test piece top surface.

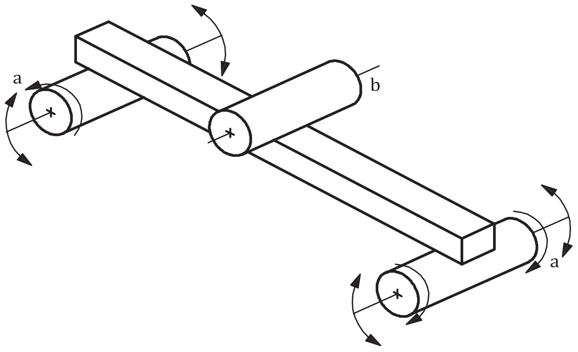


b) Fully articulating

Key

a The two support bearings are free to roll outwards.

b The middle bearing does not roll, but it can articulate to follow the test piece top surface. Support bearings can articulate to follow the test piece surface.



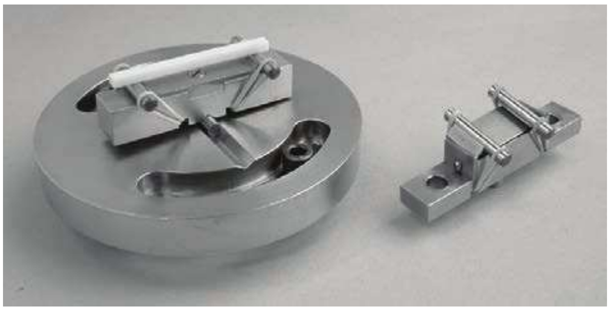
c) Fully articulating (alternative design)

Key

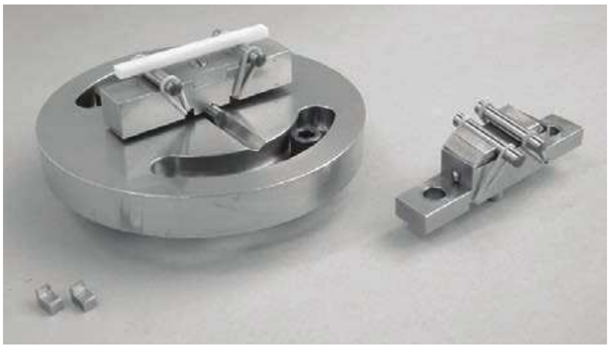
a The two support bearings are free to roll outwards, and they can articulate to follow the test piece surface.

b The middle bearing does not roll and does not articulate.

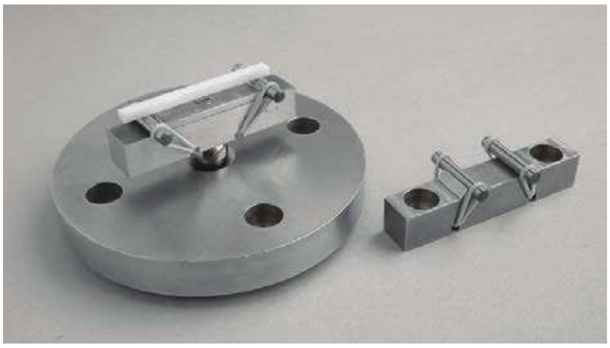
Figure B.3 — Three-point flexure fixture



a) Inner pair of bearing rollers (on the right) are parallel and articulate together as a pair in a cradle assembly



b) Same fixture as shown in a), but with inserts on the right to convert the inner span to 10 mm and a new insert on the bottom piece on the left to convert the outer span to 30 mm



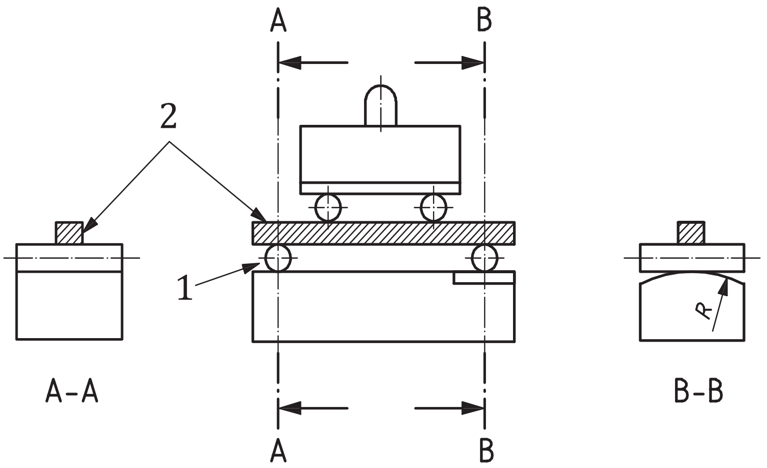
c) Alternative design with a ball on the bottom for articulation

NOTE 1 For a), the bottom outer bearing rollers (on the left) are parallel. Left-to-right articulation is carried out by a pivot underneath the bottom base. This shows a 20 mm × 40 mm span combination.

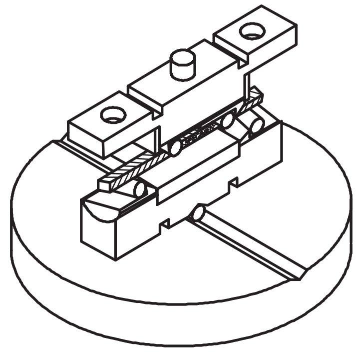
NOTE 2 For b), the two small pieces on the bottom left are attached to the back of the fixture to hold the test piece in the correct front-to-back position. They may be used with either 20 mm × 40 mm or 10 mm × 30 mm configurations.

NOTE 3 For c), the bottom base pivots on the ball that gives left-to-right and front-to-back articulation. The outer span bearings (on the left) articulate as a pair such as that shown in Figure B.1 b). The inner pair of rollers (on the right) are parallel and are fixed to the testing machine.

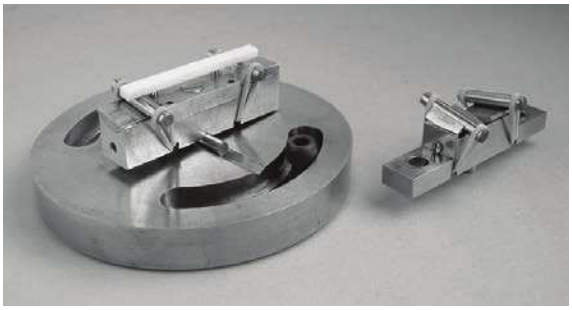
Figure B.4 — Examples of semi-articulated type fixtures



a) Schematic of a fixture with rounded base and top supports



b) Schematic of a fixture with special cradles to hold the bearing rollers



c) Photograph of the fixture shown in b)

Key

1 bearing which does not articulate

2 test piece

NOTE 1 For a), the bearing rollers may tilt or articulate to match the test piece surfaces. One of the four bearings (on the left) does not articulate. Shoulders or positioning stops for the bearing rollers are not shown for simplicity. Elastic bands, low-stiffness springs or magnets may hold the bearing rollers in their correct initial positions.

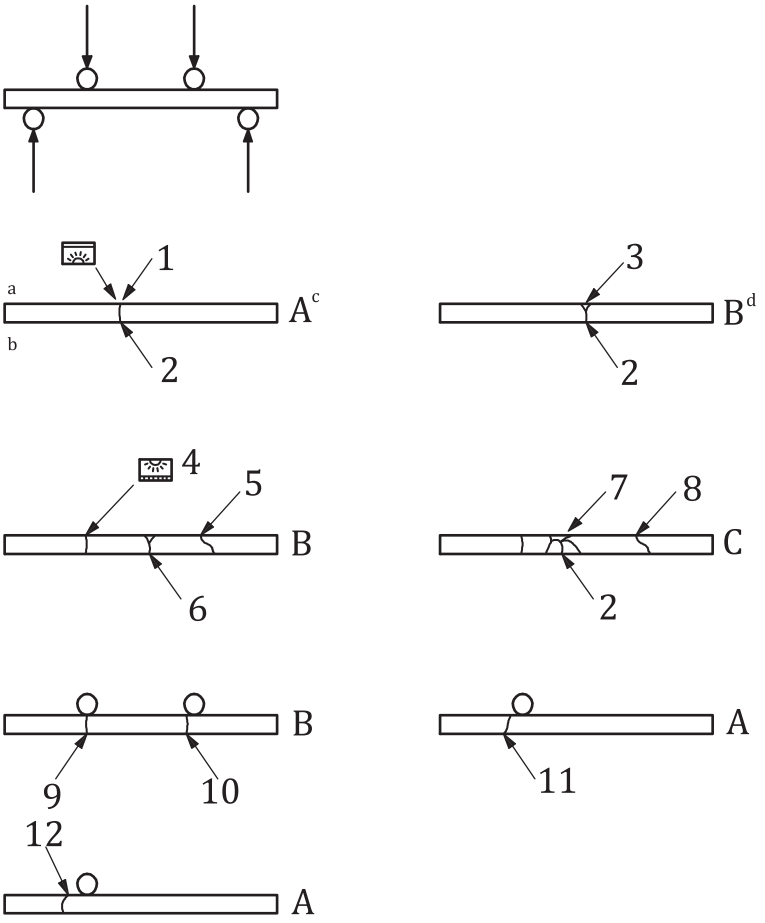
NOTE 2 For b), the cradles rotate about an axis that is on the test piece surface so that the test piece does not shift when the cradles articulate or tilt.

Figure B.5 — Examples of fully articulating fixtures

Annex C
(informative)

Typical fracture patterns in ceramic test pieces

Low-strength ceramics, which have a low energy level at fracture, typically break into only two pieces. Medium- to high-strength ceramics break into more pieces. Fractographic analysis can assist in determining the primary fracture origin. References [1] or [3] may be consulted for further guidance.



Key

1 compression curl

2 origin — fracture surface is perpendicular to the tensile surface

3 crack branch and double compression curl leading to a Y-shaped pattern (upper fragment is not important and can be discarded)

4 secondary fracture caused by the elastic release wave's reflection off the end faces (this often occurs at the loading bearings; note that the fracture mirror is on what was originally the compression side)

5 secondary break — often at an angle

6 primary fracture — origin

7 crack branching and curving back to the tensile face

8 secondary break

9 secondary fracture at a loading bearing

10 primary fracture — origin close to but not directly at a loading bearing

11 break at or near a loading bearing (beware of misalignments or twisting errors and note the angle to the tensile surface)

12 primary fracture outside the gauge length (legitimate due to severe flaw)

A low-energy failure

B medium- to high-energy failure

C high-energy failure

[a] Compression.

[b] Tension.

[c] Fracture surface is perpendicular to the tensile surface.

[d] Upper fragment is not important and can be discarded.

Figure C.1 — Typical fracture patterns in ceramic test piece

Annex D
(informative)

Chamfer correction factors

Flexural strengths should be corrected for the presence of the corner chamfers if the chamfers are oversized. Chamfers or rounded edges cause an underestimate of the true maximum flexural strength.

The maximum stress in a flexure test piece is customarily calculated from simple beam theory, with the assumption that the test piece has a rectangular cross-section. The test piece chamfers reduce the second moment of inertia, I, of the test piece cross-section about the neutral axis. For a perfect rectangular cross-section, $I = (bd^3)/12$ where b is the width and d is the thickness. For a rectangular cross-section with four chamfered edges of size c, the adjusted moment of inertia from Reference [2] is

$$I = \frac{bd^2}{12} - \frac{c^2}{9}\left(c^2 + \frac{1}{2}(3d - 2c)^2\right) \qquad \text{(D.1)}$$

where the second term on the right-hand side shows the reduction due to the chamfers. A similar formula for rounded edges in available in Reference [2].

If the chamfers or edge rounds are larger than those specified in Figure 3 (c_{max} = 0,15 mm for chamfers or R_{max} = 0,20 mm for rounded edges), then the flexural strengths should be corrected. The average chamfer size for a test piece may be used. The most accurate results may be obtained by measuring each test piece, but for many applications, an approximate chamfer size based on a sample of five test pieces may be adequate.

The correct flexural strength, σ_{f}, may be obtained by multiplying the apparent flexural strength, σ'_{f}, (calculated on the assumption that the cross-section is a simple rectangle) by a correction factor, *Cor.*

$$\sigma_{\mathrm{f}} = Cor \cdot \sigma'_{\mathrm{f}} \qquad \text{(D.2)}$$

Correction factors, *Cor,* for four chamfers or rounded edges are listed in Table D.1.

The chamfer geometry is shown in Figure D.1.

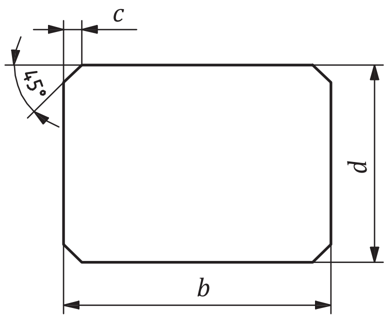


Figure D.1 — Chamfer geometry

Table D.1 — Correction factor, *Cor*, for four chamfers

C mm	**Correction factor**, *Cor* $b = 4$ mm, $d = 3$ mm
0,080	1,003 1
0,090	1,003 9
0,100	1,004 8
0,110	1,005 8
0,120	1,006 9
0,130	1,008 0
0,140	1,009 3
0,150	1,010 6
0,160	1,012 1
0,170	1,013 6
0,180	1,015 2
0,190	1,016 9
0,200	1,018 6
0,210	1,020 5
0,220	1,022 4
The bold line below $c = 0{,}150$ marks the limiting size for chamfers specified in 6.2.6 and Figure 3.	

The rounded-edge geometry is shown in Figure D.2.

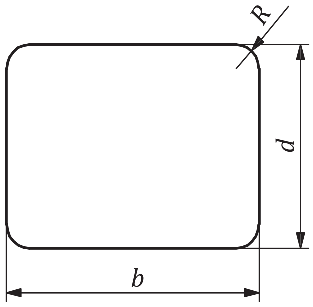


Figure D.2 — Rounded-edge geometry

Table D.2 — Correction factor, *Cor*, for four rounded edges

R mm	**Correction factor**, *Cor* $b = 4$ mm, $d = 3$ mm
0,080	1,001 3
0,090	1,001 7
0,100	1,002 1
0,110	1,002 5
0,120	1,003 0
0,130	1,003 5
0,140	1,004 1
0,150	1,004 6
0,160	1,005 3
The bold line below $c = 0{,}200$ marks the limiting size for rounded edges specified in 6.2.6 and Figure 3.	

Table D.2 *(continued)*

R mm	**Correction factor**, *Cor* *b* = 4 mm, *d* = 3 mm
0,170	1,005 9
0,180	1,006 6
0,190	1,007 4
0,200	1,008 2
0,210	1,009 0
0,220	1,009 8
0,230	1,010 7
0,240	1,011 6
0,250	1,012 6
0,260	1,013 6
0,270	1,014 6
The bold line below *c* = 0,200 marks the limiting size for rounded edges specified in 6.2.6 and Figure 3.	

Annex E
(informative)

Weibull scaling factors

Several fixture types and test piece sizes are included in this International Standard. Strengths may be converted from one size and testing configuration to other sizes and testing configurations by Weibull strength scaling.[5] The smaller 3 mm × 4 mm × ≥ 35 mm test pieces tested on 10 mm × 30 mm fixture spans will produce greater strengths than the 3 mm × 4 mm × ≥ 45 mm test pieces tested on the 20 mm × 40 mm fixture spans. If strength is controlled by a single flaw type that is volume distributed, and the strength distribution may be modelled by a Weibull two parameter distribution, then

$$\frac{\bar{\sigma}_1}{\bar{\sigma}_2} = \left(\frac{V_{E2}}{V_{E1}}\right)^{1/m} \tag{E.1}$$

where

$\bar{\sigma}_1$ is the mean strength of test piece/fixture type 1;

$\bar{\sigma}_2$ is the mean strength of test piece/fixture type 2;

V_{E1} is the effective volume of test piece type 1;

V_{E2} is the effective volume of test piece type 2.

The effective volume of the four-point test-piece-fixture configurations in this International Standard are

$$V_{E,4,30} = V\frac{(m+3)}{6(m+1)^2} \tag{E.2}$$

$$V_{E,4,40} = V\frac{(m+2)}{4(m+1)^2} \tag{E.3}$$

where

V is the total volume of the test piece within the outer loading span

(either $V = 3\text{ mm} \times 4\text{ mm} \times 30\text{ mm} = 360\text{ mm}^3$, or

$V = 3\text{ mm} \times 4\text{ mm} \times 40\text{ mm} = 480\text{ mm}^3$);

m is the Weibull modulus.

then

$$\frac{\bar{\sigma}_{4,30}}{\bar{\sigma}_{4,40}} = \left(\frac{V_{E,4,40}}{V_{E,4,30}}\right)^{1/m} = W \tag{E.4}$$

Table E.1 lists values of W for typical Weibull moduli.

Table E.1 — W versus m for Weibull volume or surface scaling

	m										
	5	6	7	8	9	10	12	15	20	25	30
W	1,118	1,101	1,088	1,078	1,070	1,063	1,053	1,043	1,033	1,027	1,022

If strength is controlled by a single flaw type that is surface distributed, and the strength distribution may be modelled by a Weibull two parameter distribution, then

$$\frac{\bar{\sigma}_1}{\bar{\sigma}_2} = \left(\frac{S_{\mathrm{E2}}}{S_{\mathrm{E1}}}\right)^{1/m} \tag{E.5}$$

where

$\bar{\sigma}_1$ is the mean strength of test piece/fixture type 1;

$\bar{\sigma}_2$ is the mean strength of test piece/fixture type 2;

S_{E1} is the effective surface of test piece type 1;

S_{E2} is the effective surface of test piece type 2.

The effective surfaces of the four-point test-piece-fixture configurations in this International Standard are

$$S_{\mathrm{E,4,30}} = L\left[h + b\left(m+1\right)\right] \frac{m+3}{3(m+1)^2} \tag{E.6}$$

$$S_{\mathrm{E,4,40}} = L\left[h + b\left(m+1\right)\right] \frac{m+2}{2(m+1)^2} \tag{E.7}$$

where

L is the outer loading span (30 mm or 40 mm);

b is the test piece width, 4 mm;

h is the test piece height, 3 mm;

m is the Weibull modulus.

then

$$\frac{\bar{\sigma}_{4,30}}{\bar{\sigma}_{4,40}} = \left(\frac{S_{\mathrm{E,4,40}}}{S_{\mathrm{E,4,30}}}\right)^{1/m} = W \tag{E.8}$$

The factor, W, for surface strength scaling *is identical* to the factor, W, for volume strength scaling in Table E.1.

NOTE 1 This surprising outcome can be confirmed by algebraic manipulation of the above formulae. It is only true for certain specific test-piece-fixture configurations such as those specified in this International Standard.

NOTE 2 For converting strengths, it is not necessary to know whether the fracture origins are surface or volume distributed flaws when using the test-piece-fixture configurations in this International Standard.

Bibliography

[1] ASTM C1322-96a, *Standard Practice for Fractography and Characterization of Fracture Origins in Advanced Ceramics*

[2] Baratta F.I., Quinn G.D., Matthews W.T. *Errors Associated with Flexural Testing of Brittle Materials*, U.S. Army Technical Report, MTL TR 87-35 (available from G.D. Quinn, NIST - Ceramics Division, Gaithersburg, MD 20899, U.S.A.)

[3] MIL HDBK 790, *Fractography and Characterization of Fracture Origins in Advanced Ceramics*, U.S. Army Military Handbook, 1 July 1992 (available from U.S. Army Research Laboratory, Materials Directorate, Aberdeen Proving Ground, MD 21005, U.S.A.)

[4] Quinn G.D., & Morrell R. Design Data for Engineering Ceramics: A Review of the Flexural Test. *J. Am. Ceram. Soc.* 1991, **74** (9) pp. 2037–2066

[5] Quinn G.D. Weibull Strength Scaling for Standardized Rectangular Flexure Test pieces. *J. Am. Ceram. Soc.* 2003, **86** (3) pp. 508–510

ICS 81.060.30

Price based on 28 pages