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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

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 14704 was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

Annex B forms a normative part of this International Standard. Annexes A and C are for information only.

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

1 Scope

This International Standard describes the method of test for determining the flexural strength of monolithic fine ceramics and whisker- or particulate-reinforced ceramic composites at room temperature. Flexural strength is one measure of the uniaxial strength of a fine ceramics. This test method may be used for materials development, quality control, characterization and design data generation purposes.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 7500-1:1999, *Metallic materials — Verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Verification and calibration of the force-measuring system.*

ISO 3611:1978, *Micrometer callipers for external measurement.*

3 Terms and definitions

For the purpose of this International Standard, 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 specimen is loaded equally by two bearings symmetrically located between two support bearings

[see Figure 1 a) and b)]

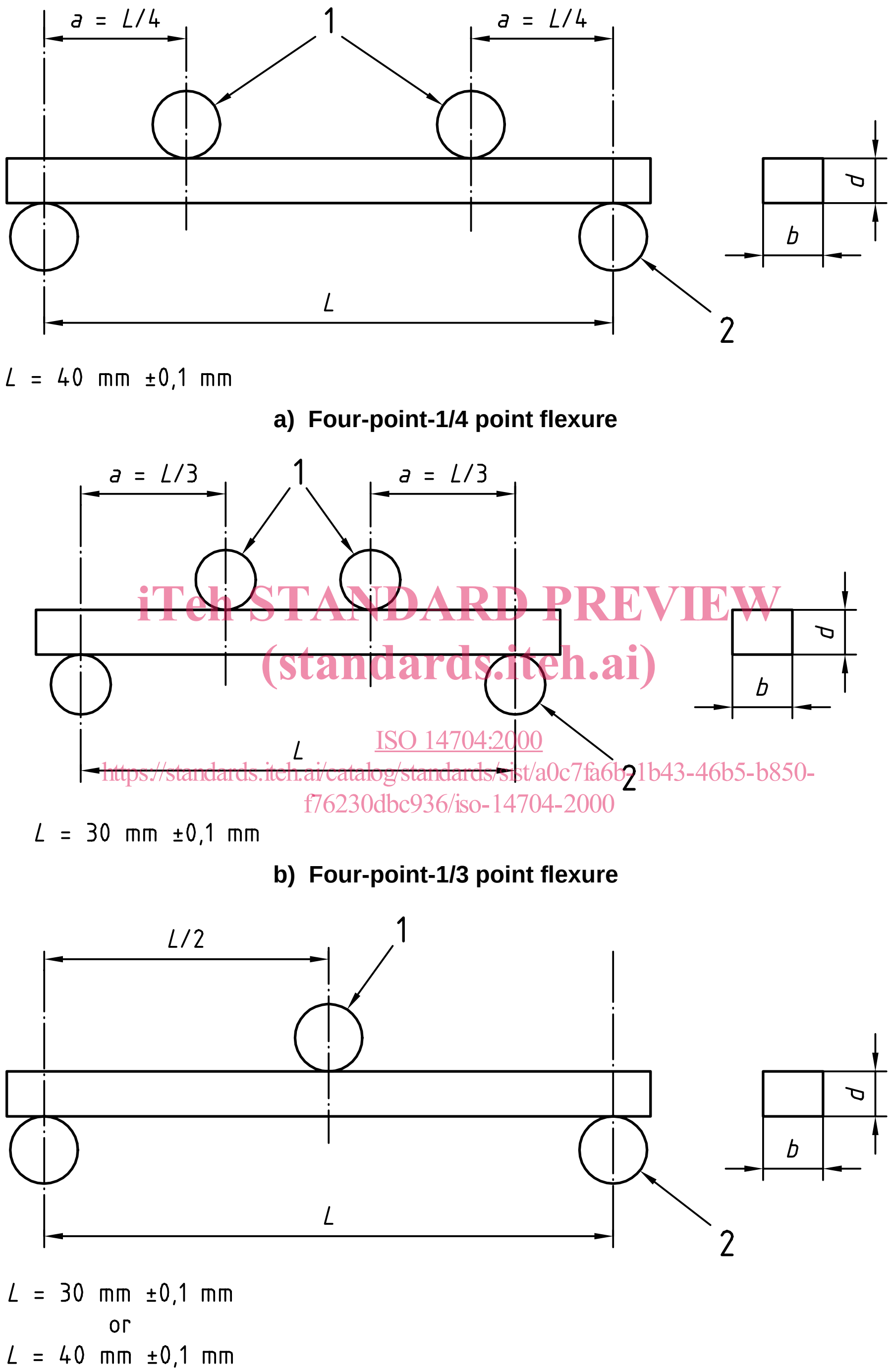
NOTE The bearings may be cylindrical rollers or cylindrical bearings.

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

[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

[see Figure 1 b)]



a) Four-point-1/4 point flexure

b) Four-point-1/3 point flexure

c) Three-point flexure

Key

1 Loading bearings

2 Support bearing

NOTE Four-point flexure is usually preferred since a large amount of material is exposed to the maximum stress (see annex A for more information).

Figure 1 — Flexural test configurations

4 Principle

A beam 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 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 Testing machine

A suitable testing machine capable of applying a uniform cross-head speed shall be used. The testing machine shall be in accordance with ISO 7500-1:1999, 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 fixtures shall either be semi-articulating or fully-articulating depending upon the condition of the specimens. If the specimens meet the parallelism requirement of 6.1, then semi-articulating fixtures may be used, otherwise the fully-articulating fixtures shall be used. Fully-articulating fixtures also may be used with machined specimens.

NOTE 1 Specimens that are machined will normally have flat and parallel surfaces. As-fired, heated treated or oxidized specimens often do not meet the parallelism requirements. Twisting of the specimen can cause severe errors in the strength calculation, unless a fully-articulating fixture is used. The purpose of articulation is to ensure that the bearings have a uniform and even contact with the specimen surface.

NOTE 2 A fully-articulating fixture has bearings or rollers that are free to roll to eliminate friction. The bearings articulate independently to match the specimen surface. See Figures B.1 and B.2.

NOTE 3 A semi-articulating fixture has bearings or rollers that are free to roll. The bearings articulate in pairs to match the specimen surface. See Figures B.1 and B.2.

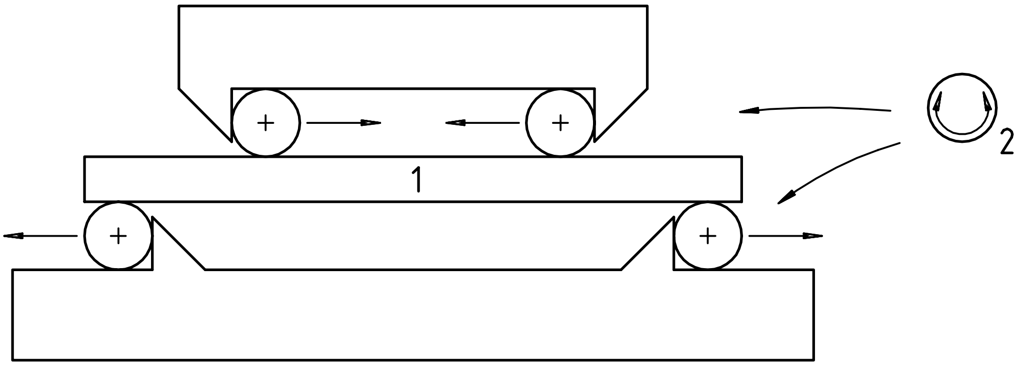
5.2.2 Bearings

Specimens 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 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 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 specimen thickness (d). Diameters between 4,5 mm and 5 mm are recommended. The bearings shall have a smooth surface and shall have a diameter uniform to ± 0,015 mm. The bearings shall be free to roll in order to eliminate friction. In a 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 a 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 may 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.

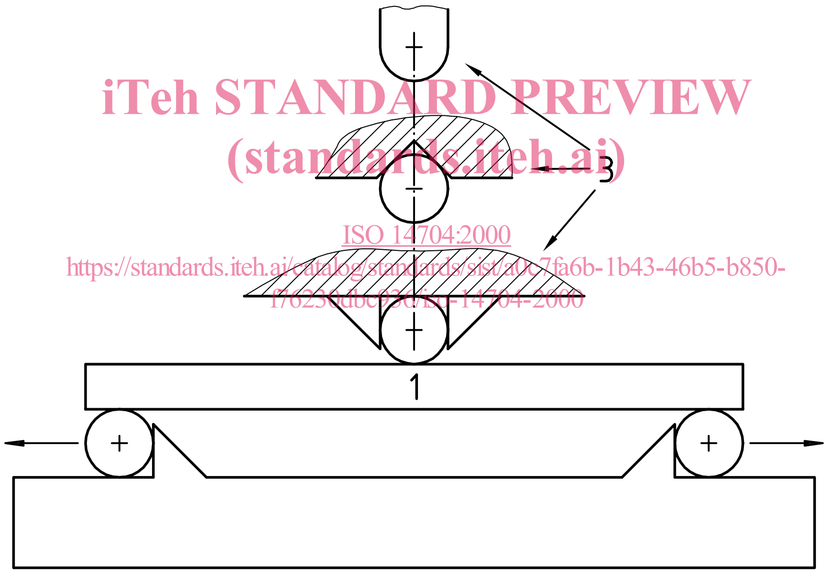
NOTE 2 The bearing diameter is specified on the basis of competing requirements. The bearings should not be so large as to cause excessive change in the moment arm as a specimen deflects as this can create errors from contact-point tangency shift. On the other hand, the bearings should not be so small as to create excessive wedging stresses in the specimen or create contact stresses that damage the fixture.

NOTE 3 The bearing hardness and stiffness requirements and guidelines are intended to ensure that specimens with strength up to 1400 MPa (or 2000 MPa) and elastic moduli as high as 500 GPa can be tested without damaging the fixture. Higher strength or stiffer ceramic specimens may 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.



The four bearings shall be free to roll.

a) Four-point flexure



The two outer bearings are free to roll outwards, but the middle bearing shall be non-rolling.

b) Three-point flexure

Key

1 Specimen

2 Alternative rolling bearings

3 Alternative loading bearing arrangements

Figure 2 — Schematic representation of fixtures

5.2.3 Four-point fixture: semi-articulating

Figure B.1 a) shows the actions of the bearings in this fixture. All four bearings shall be free to roll. 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. The inner bearings shall be supported independently of the outer bearings. All four bearings shall rest uniformly and evenly across the specimen surface. The fixture shall be designed to apply equal load to all four bearings.

5.2.4 Four-point fixture: fully-articulating

Figure B.1 b) shows the actions of the bearings in this fixture. All four bearings shall be free to roll. One bearing need not articulate. The other three bearings shall articulate independently to follow the specimen surface. All four bearings shall rest uniformly and evenly across the specimen surface. The fixture shall apply equal load to all four bearings.

5.2.5 Three-point fixture: semi-articulating

Figure B.2 a) shows the actions of the bearings in this fixture. The two support (outer) bearings shall be free to roll outwards. The middle bearing shall be fixed and not free to roll. 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 specimen surface, or the middle bearing shall articulate to follow the specimen surface. All three bearings shall rest uniformly and evenly across the specimen surface. The fixture shall be designed to apply equal load to the two outer bearings.

5.2.6 Three-point fixture: fully-articulating

Figure B.2 b) and c) show the actions of the bearings in this fixture. The two support (outer) bearings shall be free to roll outwards. The middle bearing shall not roll. Any two of the bearings shall be capable of articulating independently to follow the specimen surface. The fixture shall be designed to apply equal load to the two outer bearings.

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 may 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 may be measured to the nearest 0,1 mm using a travelling microscope or other suitable device. The spans may 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.

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

6 Test specimens

6.1 Specimen size

6.1.1 Machined specimens

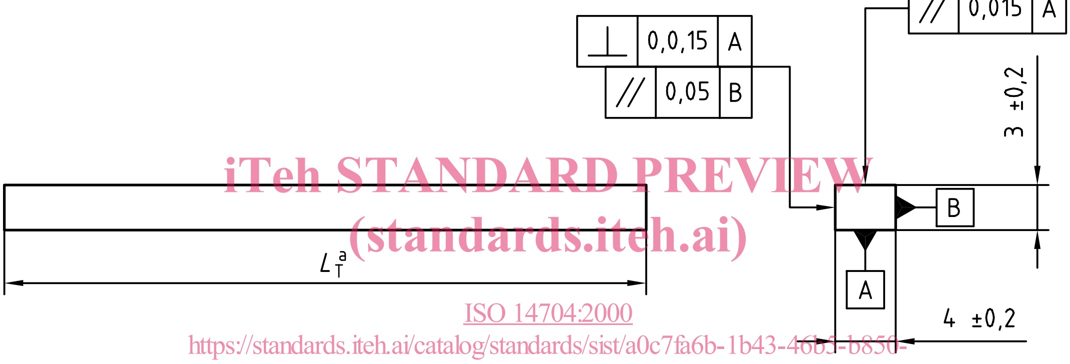
Specimen dimensions are shown in Figure 3. Cross-sectional tolerances shall be ± 0,2 mm. The parallelism tolerance on opposite longitudinal faces is 0,015 mm.

6.1.2 As-fired or heat treated specimens

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

Dimensions in millimetres

Edge chamfers at (0,12 ± 0,03) mm × 45° ± 5°
or rounded at (0,15 ± 0,05) mm



[a] $L_T \geqslant$ 35 mm for 30 mm test fixtures but $\geqslant$ 45 mm for 40 mm test fixtures.

Figure 3 — Standard test specimen

6.2 Specimen preparation

6.2.1 General

This International Standard allows several options for specimen preparation. In all cases, the end faces of the specimen 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. 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 specimens can introduce machining flaws (especially microcracks beneath the specimen surface) which may 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) may or may not negate machining damage introduced from prior, coarser machining steps.

6.2.2 As-fired

The flexure specimen 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 specimen with an as-fired surface. An edge chamfer or rounding is recommended and can be made before sintering.

NOTE 1 As-fired specimens 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.

NOTE 2 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 (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 may be used. The 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 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 report shall include details of the procedure, especially the wheel bonding (resin, metal, vitreous glass, other) and the material removed per pass. The long edges of the specimen 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 may be suitable for some materials. Alternately, some very brittle ceramics may require a more conservative preparation.

6.2.5.1 Specimens shall be ground in the longitudinal direction as shown in Figure 4.

6.2.5.2 All grinding shall be done with an ample supply of filtered coolant in order to keep the workpiece 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), effecting a depth of cut not exceeding 0,03 mm per pass. Alternately, 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), effecting 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 specimen surfaces in the fine finishing step. The direction of machining shall be parallel to the specimen's long axis.

6.2.5.6 The specimen final dimensions shall be in accordance with 6.1.1 and Figure 3.

6.2.6 Handling of specimens

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