INTERNATIONAL STANDARD

ISO 14704

Second edition
2008-02-01

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 14704:2008(E)

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Published in Switzerland

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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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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

This second edition cancels and replaces the first edition (ISO 14704:2000) and the technical corrigendum (ISO 14704:2000/Cor. 1:2004), which have 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. 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 referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

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

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 specimen is loaded equally by two bearings symmetrically located between two support bearings

See Figure 1 a) and b).

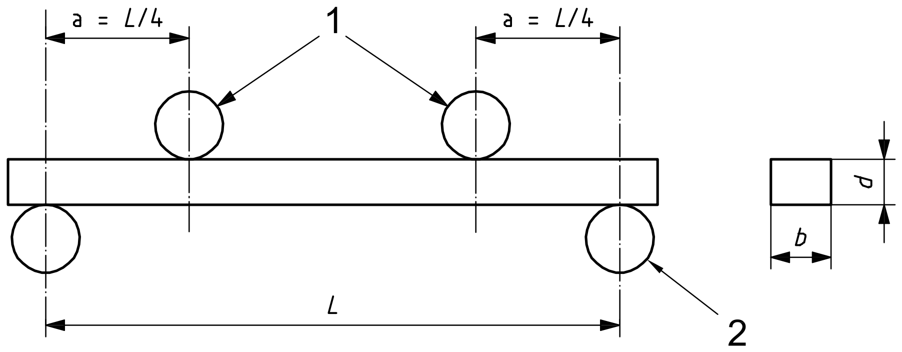
NOTE The bearings may be cylindrical rollers or cylindrical bearings.

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).

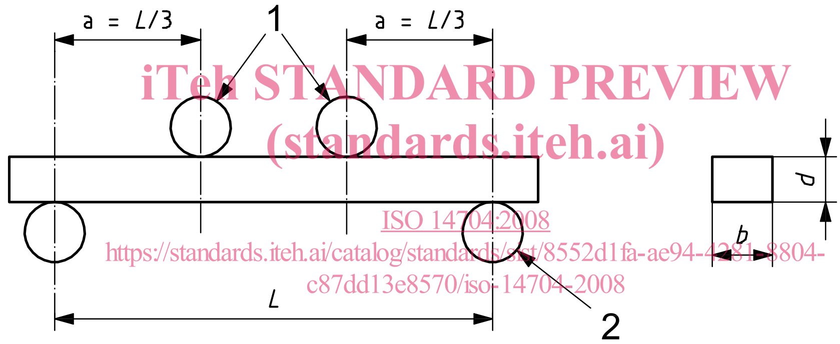
3.4
four-point-1/3 point flexure
specific configuration of four-point flexural strength testing where the inner bearings are situated one-third of the support span away from the two outer bearings

See Figure 1 b).



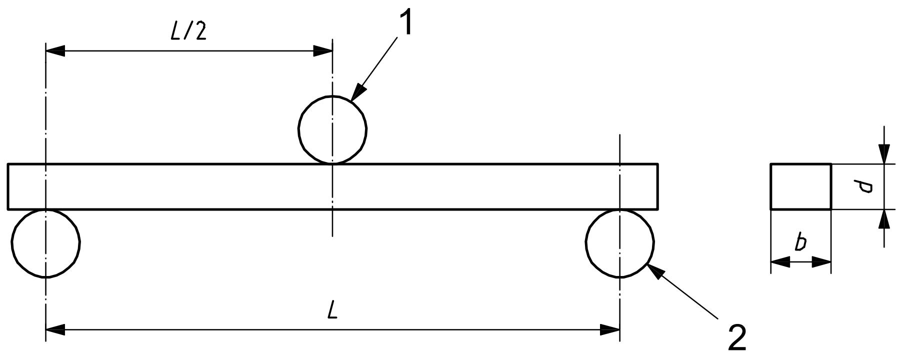
L = 40 mm ± 0,1 mm

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 L = 40 mm ± 0,1 mm

c) Three-point flexure

Key

1 loading bearings

2 support bearing

Figure 1 — Flexural test configurations

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

3.6
fully articulating fixture
test fixture designed to apply uniform and even loading to specimens that may have uneven, non-parallel or twisted surfaces

3.7
three-point flexure
configuration of flexural strength testing where a specimen is loaded at a location midway between two support bearings

See Figure 1.

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).

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 equipped for recording the peak load applied to the test piece. The testing machine shall be in accordance with ISO 7500-1:2004, 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 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 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 specimen surfaces and apply 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 on as-machined specimens. A semi-articulating fixture has pairs of upper and lower bearings that articulate to match the specimen surfaces, as shown in Figures B.2 and B.3. Fully articulated fixtures have more moving parts and are necessary for specimens 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 specimens, since uneven loading can cause twisting and severe errors. A fully articulating fixture may also be used with machined specimens.

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 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 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 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 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 2 The bearing hardness and stiffness requirements and guidelines are intended to ensure that specimens 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 specimens 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 specimen surface. All four bearings shall rest uniformly and evenly across the specimen 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 specimen surface. All four bearings shall rest uniformly and evenly across the specimen 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 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. The two support (outer) bearings shall be free to roll outwards. The middle bearing shall be fixed and not free to roll.

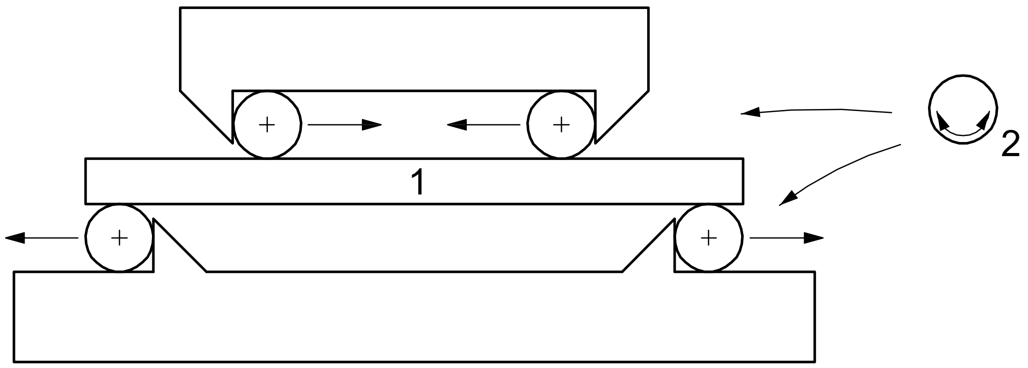
5.2.6 Three-point fixture: fully articulating

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 specimen 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

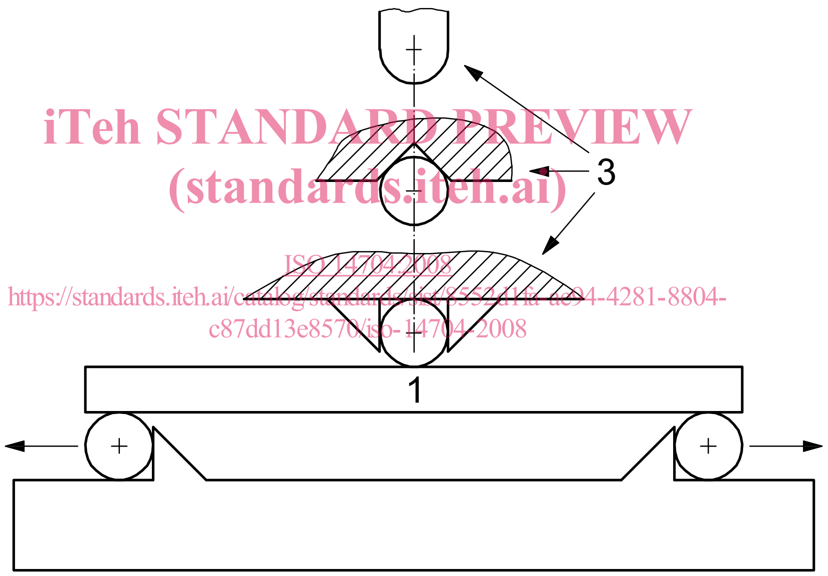
The bearings shall be positioned so that the spans are accurate to within ± 0,1 mm. The middle bearing for the three-point fixture shall be positioned midway between the outer bearings to within ± 0,1 mm. The inner bearings for the four-point fixture shall be centred over the outer bearings to within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



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 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 specimen 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 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

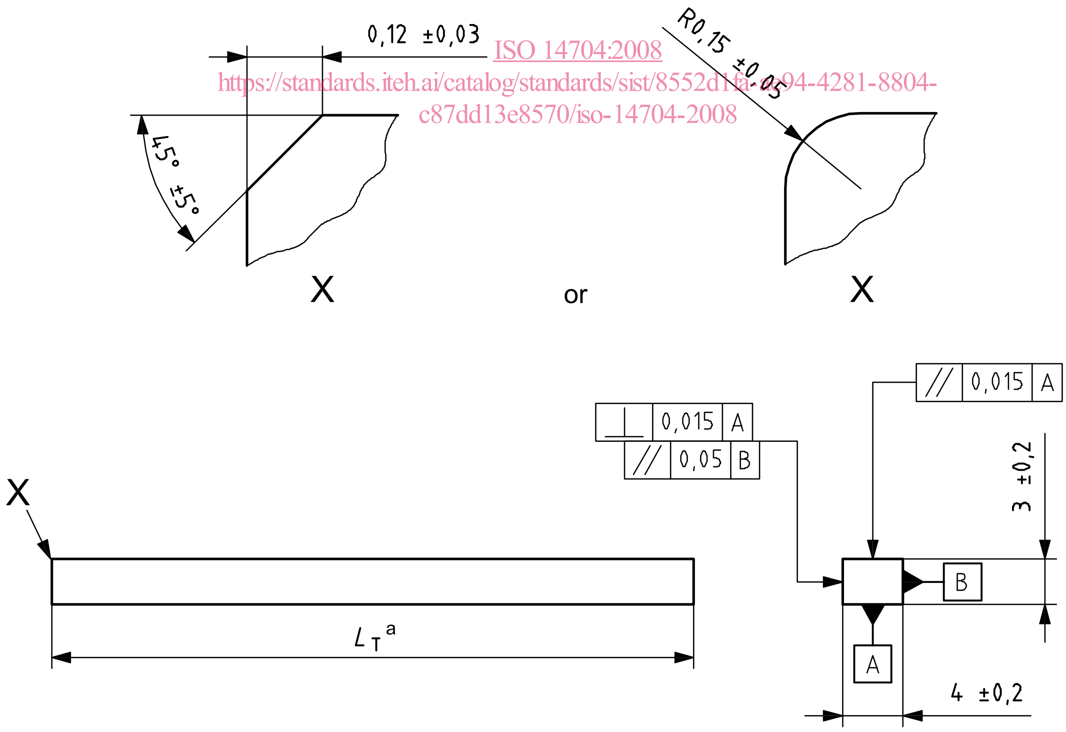
Specimen dimensions are shown in Figure 3. Cross-sectional tolerances shall be ± 0,2 mm. The parallelism tolerance on opposite longitudinal faces is 0,015 mm.

NOTE The terms “specimen” and “test piece” are used interchangeably in this International Standard.

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 test report.

Dimensions in millimetres



a $L_T \geqslant 35$ mm for 30 mm test fixtures.

$L_T \geqslant 45$ 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 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 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.

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.

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 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 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 wheel bonding (resin, metal, vitreous, 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 can be suitable for some materials. Alternatively, some very brittle ceramics can require a more conservative preparation.

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

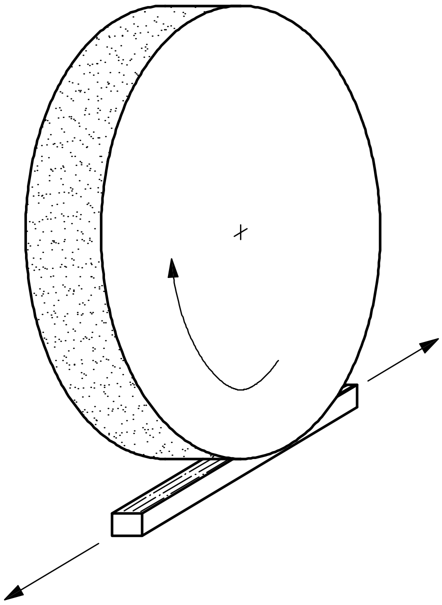


Figure 4 — Surface grinding parallel to the specimen longitudinal axis

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 specimen surfaces in the fine-finishing step. The direction of machining shall be parallel to the specimen'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 specimen cross-section. Annex D may be consulted for this correction.

6.2.5.6 The final dimensions of the specimen 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 (3 to 5 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 centre. 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.

