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

*Céramiques techniques — Méthode d'essai de résistance à la flexion
des céramiques monolithiques à température élevée*



Reference number
ISO 17565:2003(E)

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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 17565 was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for flexural strength of monolithic ceramics at elevated 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 elevated temperature. Flexural strength is one measure of the uniaxial strength of 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:—¹⁾, *Metallic materials — Verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Verification and calibration of the force-measuring system*

ISO 14704:2000, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for flexural strength of monolithic ceramics at room temperature*

IEC 60584-1:1995, *Thermocouples — Part 1: Reference tables*

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

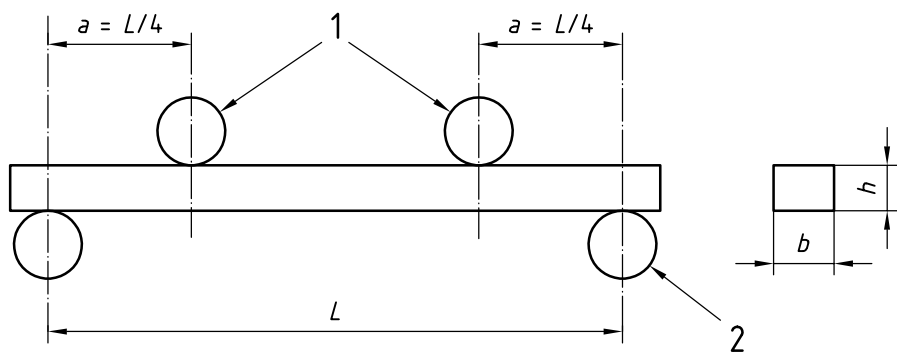
four-point flexure

configuration of flexural strength testing where a test piece is equally loaded by two bearings symmetrically located between two support bearings

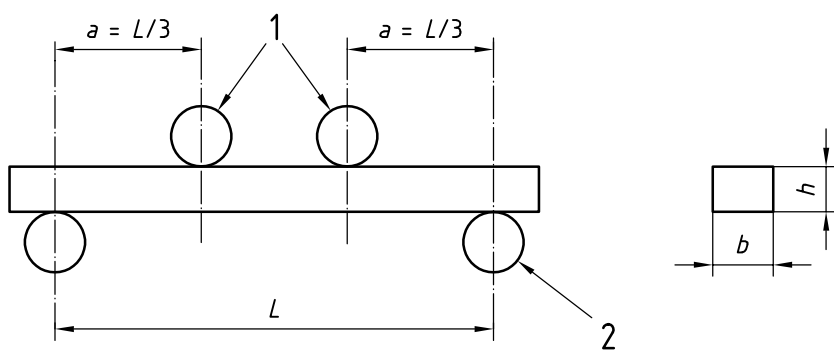
See Figure 1.

NOTE The bearings may be cylindrical rollers or cylindrical bearings.

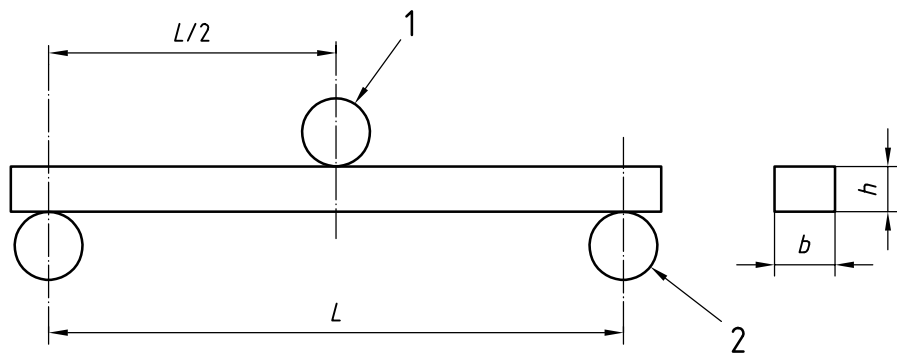
1) To be published. (Revision of ISO 7500-1:1999)



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

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

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).

3.5**three-point flexure**

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

See Figure 1 c).

NOTE Four-point flexure is usually preferred since a larger amount of material is exposed to the maximum stress. See Bibliography 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 a measure of the uniaxial tensile strength of a ceramic. The material is assumed to be isotropic and linearly-elastic. Testing is performed at elevated temperature in air or inert atmosphere. Load is monitored versus displacement or time in order to confirm that the beam test piece fractured under elastic conditions. This International Standard is an elevated-temperature adaptation of ISO 14704. See Annex A for additional information on this test method.

5 Apparatus**5.1 Testing machine**

A suitable testing machine capable of applying a uniform crosshead speed shall be used. The testing machine shall be in accordance with ISO 7500-1:— Class 1 with an accuracy of 1 % of indicated load at fracture.

5.2 Heating device**5.2.1 General**

The furnace shall be capable of heating the test fixture and test piece as well as maintaining a uniform and constant temperature during the strength test. The furnace may have an air, inert gas or vacuum environment, as required. If an inert gas or vacuum chamber is used, and it is necessary to transmit the load through a seal, bellows or a fitting, it shall be verified that load losses or errors are less than 1 % of the expected fracture loads. The furnace may be designed so that test pieces are loaded into either hot or cold furnaces.

5.2.2 Test piece temperature stability

The furnace shall be controlled by a device for maintaining a constant temperature within ± 2 °C or better within the working space of the furnace, during the time that the test piece is loaded to fracture.

5.2.3 Test piece temperature uniformity

The furnace shall be capable of maintaining the test piece temperature uniform. It shall previously be determined that the temperature of the test piece shall not vary more than 10 °C over its length after a 15 min hold time at the required test temperature.

5.2.4 Furnace heating rate

The furnace control device shall also be capable of controlling the heating rates of the furnace and preventing temperature overshoots.

5.2.5 Furnace stability

The time for the system to reach thermal equilibrium at test temperature shall be determined for the test temperature to be used.

5.3 Temperature measuring and indicating instruments

5.3.1 General

Thermocouple temperature measuring equipment shall have a resolution of at least 1 °C and an accuracy of 5 °C or better. Optical pyrometers, if used, shall have a resolution of at least 5 °C and an accuracy of 5 °C or better.

NOTE 1 Resolution should not be confused with accuracy. Beware of instruments that have a resolution (read out) of 1 °C, but have an accuracy of only 10 °C; e.g., an instrument with a 1 % accuracy would only be accurate to ± 12 °C at 1 200 °C.

NOTE 2 Thermocouple temperature measuring instruments typically approximate the temperature-electromotive force (EMF) tables, but with a few degrees error.

5.3.2 Thermocouples

Thermocouples in accordance with IEC 60584-1 shall be used. The thermocouples shall exhibit low thermal inertia (the diameter of the wires shall not be greater than 0,5 mm). The thermocouples shall have a sufficient length within the furnace (with respect to heat conduction along the wires). The measuring thermocouple tip shall be as close as possible to or contacting the test piece.

NOTE 1 In some furnaces, a control thermocouple may be installed at a location within the furnace which is convenient for furnace control and a second measuring thermocouple may be in close proximity to the test piece.

NOTE 2 The thermocouple should not contact the test piece if there is a chance the test piece will be misaligned.

NOTE 3 The thermocouple should not contact the test piece if it will chemically react with the test piece.

5.3.3 Verification of the thermocouple temperature measuring system

Thermocouples shall be checked periodically since calibration may drift with usage or contamination.

5.3.4 Radiation pyrometers

Radiation pyrometers (thermometers) may be used in instances wherein suitable thermocouples are not available, particularly at temperatures above 1 600 °C. Pyrometers may either be of the disappearing filament type or an automatic type. Pyrometers may either be spectral (operate over a narrow wavelength band), dual wavelength (operate over two narrow wavelength bands) or total (integrate or average over all wavelengths). Radiation pyrometry requires special care in order to obtain accurate and precise results. Ensure that blackbody conditions are obtained, or correct the temperature for the actual emissivity of the test piece in its environment. The pyrometer should view the test piece at right angles to one of the four long surfaces. The target size (or viewed area) of the pyrometer shall be less than 3 mm in diameter. Corrections for radiation absorption or reflectance may be necessary if the furnace has a window. The window shall be sufficiently large such that the radiation emanating from the window completely fills the objective lens of the radiation thermometer, but the window should not be so large as to cause appreciable heat loss or thermal gradients in the furnace.

NOTE See ASTM Standard Test Methods E 452, E 639, E 1256, and BS 1041, part 5 for additional information.

5.4 Testing fixture

5.4.1 General

Three- or four-point flexure configurations shall be used as illustrated in Figures 1 and 2. The four-point-1/4-point configuration is recommended. The fixtures shall either be semi-articulating or fully-articulating as specified in ISO 14704 depending upon the condition of the test pieces. If the test pieces meet the parallelism requirements of 6.1.1 and Figure 3, either a semi-articulating fixture or fully articulating fixture may be used. If the test pieces do not meet the parallelism requirements of 6.1.1 and Figure 3, a fully-articulating fixture shall be used.

NOTE 1 Machined test pieces normally have flat and parallel surfaces and semi-articulating fixtures are completely satisfactory. On the other hand, as-fired, heated treated or oxidized test pieces often do not meet the parallelism requirements. Twisting of the test piece 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 uniform and even contact with the test piece 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 test piece surface.

NOTE 3 A semi-articulating fixture has bearings or rollers that are free to roll to eliminate friction. The bearings articulate in pairs to match the test piece surface.

It is recognized that practical limitations may restrict the design of the test fixture, and oxidation effects may restrict its function. In such cases, alternatives may be employed, but deviations from the function specified above shall be reported in the report.

5.4.2 Bearings

Test pieces shall be loaded and supported by bearings. The bearings shall be cylindrical rollers. 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, h . Diameters between 4,5 mm and 5,0 mm are recommended. The bearings shall have a smooth surface and shall have a diameter uniform to $\pm 0,015$ mm. All bearings shall be free to roll in order to eliminate friction, with the exception of the middle bearing in three point flexure (see Figure 2). For 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 should not roll.

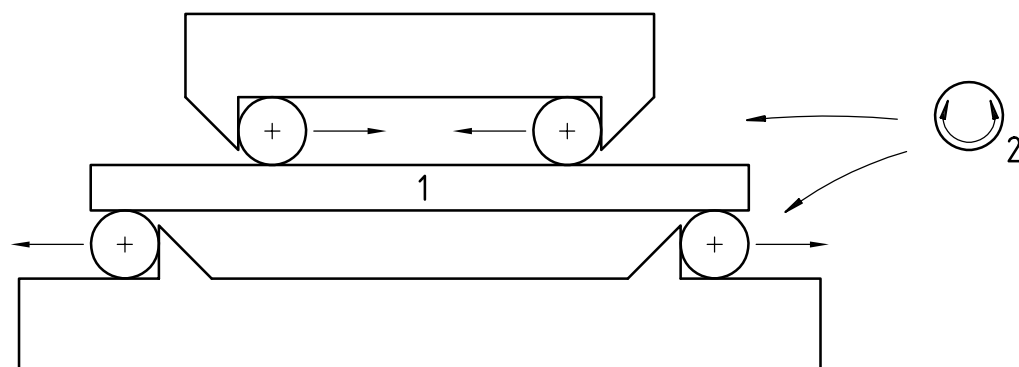
NOTE 1 Friction can cause errors in the stress calculations. The rolling action in elevated temperature fixtures is usually accomplished by using cylindrical bearings which rest on a flat surface. The bearings are free to roll on the surface as shown in Figure 2.

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 (" a " in Figure 1) as a test piece deflects which can create errors from contact-point tangency shift. On the other hand, the bearings should not be so small as to create excessive wedging or contact stresses in the test piece.

5.4.3 Four-point fixture — Semi-articulating

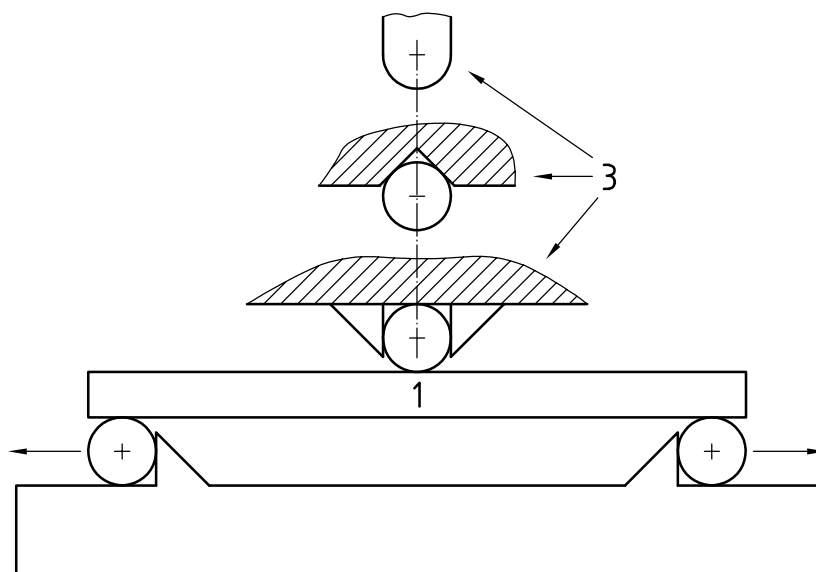
All four bearings shall be free to roll. The two inner bearings shall be parallel to each other to within 0,015 mm over their length (≥ 12 mm in accordance with 5.4.2). The two outer bearings shall be parallel to each other to within 0,015 mm over their length. The inner bearings shall be supported independently of the outer bearings. All four bearings shall rest uniformly and evenly across the test piece surface. The fixture shall be designed to apply equal loads to all four bearings.

NOTE The parallelism requirement may be met by control of the test fixture dimensions.



The four bearings shall be free to roll in four-point flexure

a) Four-point flexure



The two outer bearings are free to roll outwards in the three-point flexure, but the middle bearing shall be non-rolling

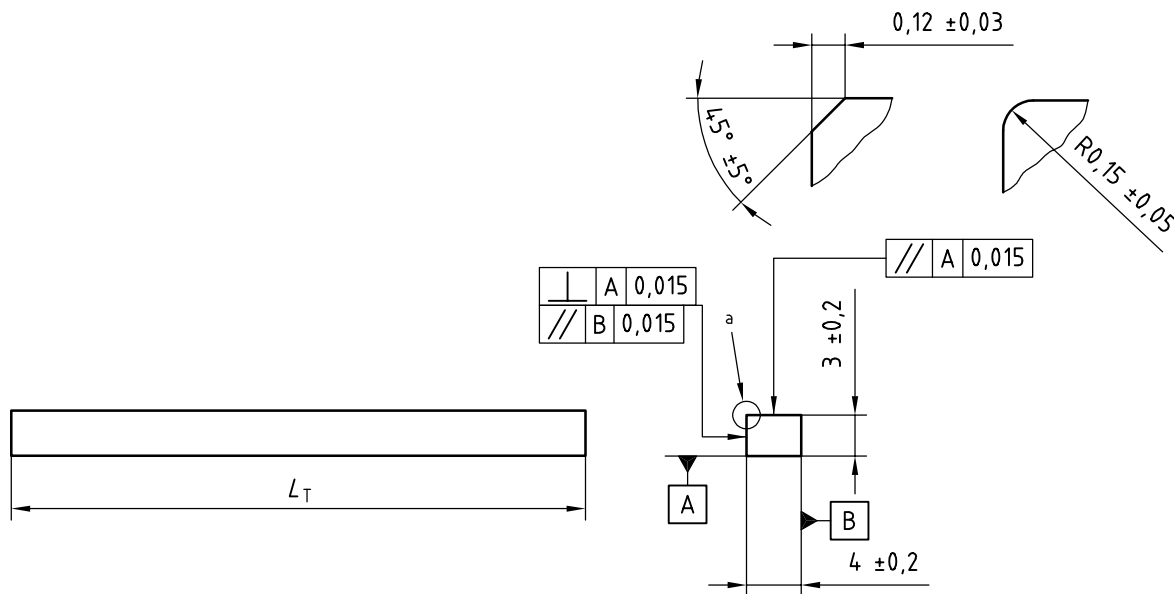
b) Three-point flexure

Key

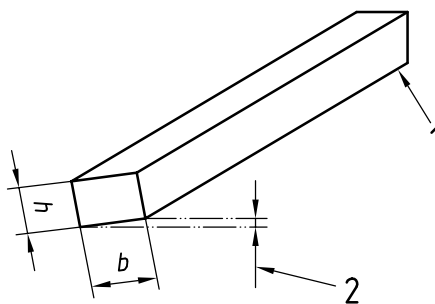
- 1 test piece
- 2 alternative rolling bearings
- 3 alternatives

Figure 2 — Schematic of semi-articulating fixtures

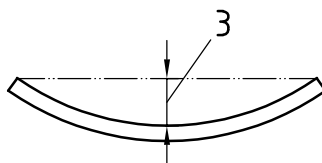
Dimensions in millimetres



a) The standard test piece



b) As-fired or heat treated test piece (see 6.1.2) — Specimen twist



c) As-fired or heat treated test piece (see 6.1.2) — Specimen bow

Key

- 1 one end on flat surface
- 2 gap
- 3 bow
- a edge chamfers or rounding

Figure 3 — Test pieces

5.4.4 Four-point fixture — Fully-articulating

All four bearings shall be free to roll. Three bearings shall articulate independently to match the test piece's surface. The fourth bearing need not articulate. All four bearings shall rest uniformly and evenly across the test piece surfaces. The fixture shall apply equal load to all four bearings.

5.4.5 Three-point fixture — Semi-articulating

The two support (outer) bearings shall be free to roll outwards. The middle bearing should be centrally located and does not need 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 5.4.2). The two outer bearings shall articulate together to match the test piece surface or the middle bearing shall articulate to match 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.

NOTE The parallelism requirement may be met by control of the test fixture dimensions.

5.4.6 Three-point fixture — Fully-articulating

The two support (outer) bearings shall be free to roll outwards. The middle bearing should not roll. Any two of the bearings shall be capable of articulating independently to match 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 loads to the two outer bearings.

5.4.7 Positioning of bearings

The bearings shall be positioned so that the spans have the prescribed dimensions to within $\pm 0,10$ mm. The middle bearing for the three-point fixture shall be positioned midway between the outer bearings to within $\pm 1,00$ mm. The inner bearings for the four-point fixture shall be centred between the outer bearings to within $\pm 0,10$ mm.

NOTE 1 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.

NOTE 2 Some fixtures have bearings that fit into square slots with a slight clearance. Of course, the clearance should be such that the possible spans are within the prescribed limits of this International Standard. Unfortunately, it is usually not possible to verify whether a bearing sits against an inner or outer shoulder, and thus it is possible that some bearings are free to roll and others are not. This can lead to the addition of unpredictable, random friction errors. Fixtures with such slots should be used with caution.

5.4.8 Fixture material

The fixture material shall be as inert as possible for the testing conditions used. The fixture shall be oxidation resistant if the testing is done in air. The fixture shall have negligible chemical reaction with and shall not contaminate the test piece. The fixture shall remain elastic over the load and temperature ranges used. The bearings may be made of a ceramic with an elastic modulus between 200 GPa and 500 GPa and a flexural strength greater than 275 GPa at elevated temperature.

NOTE 1 Bearings should be cleaned regularly. Silicon carbide papers are helpful in cleaning scale, oxidation products or chemical reaction products off the fixtures.

NOTE 2 Various grades of silicon carbide or sapphire are suitable. Hot-pressed or sintered silicon carbides with low additive content are elastic up to temperatures in excess of 1 500 °C. Siliconized silicon carbides and high purity aluminas are less expensive, but may exhibit creep deformation at temperatures over 1 200 °C. Recrystallized silicon carbides are elastic up to temperatures of 2 000 °C, but are relatively weak due to porosity. Graphites are extremely refractory but are restricted to inert atmospheres and are soft and have a low elastic modulus. Graphites may be suitable for loading rams or portions of fixtures, but they should not be used where there are concentrated loads such as at the loading bearings.

NOTE 3 In some instances it may be suitable to use bearings made of a different material than the test fixture; e.g., sintered silicon carbide bearings may be used with hot-pressed silicon carbide fixtures. The sintered silicon carbide is brittle and may have intermediate strength, but has low additive content and is highly oxidation resistant. The hot-pressed silicon carbide is much stronger but may oxidize and react with the test piece.

5.4.9 Fixture compliance

It is recommended that the compliance of the load train be characterized for the loading range and temperature used. An oversized ceramic block may be inserted into the fixture and loaded to the maximum expected breaking load at the test temperature. The load train and fixture shall be sufficiently rigid that at least 70 % of the crosshead motion is transmitted to the ceramic test pieces.

5.5 Micrometer

A micrometer such as is shown 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 anvils. 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 pieces shall be prepared in accordance with ISO 14704. Test piece dimensions are shown in Figure 3. Cross-sectional tolerances shall be $\pm 0,5$ 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. Test piece dimensions are shown in Figure 3, except that cross section sizes shall be within $\pm 0,5$ mm of the prescribed dimensions. The initial curvature of a test piece for 4-point loading shall be such that any specimen bow shall be less than 0,4 mm as shown in Figure 3 c). The bow shall be less than 0,8 mm for 3-point testing on 30 mm outer spans, or less than 1,3 mm for 3-point testing on 40 mm outer spans. In addition, the 4 mm wide surfaces which contact the bearings shall have a flatness of 0,15 mm or smaller. Deviations from the specifications in 6.1.1 and Figure 3 shall be stated in the report. Special note shall be made if the test pieces are twisted in which case an estimate of the twist angle along the length of the test piece shall be reported.

NOTE 1 Very twisted [Figure 3 b)] or bowed [Figure 3 c)] test pieces may interfere with proper test fixture articulation with an attendant loss of accuracy and precision. The intrinsic error associated with testing test pieces with an initial curvature is quite small and is usually much less than 1 % [6]. Surface reaction layers or irregularities may also interfere with proper force distribution in the test fixtures.

NOTE 2 Test piece twist may be estimated by holding one end of the test piece on a flat surface and observing the opposite end of the test piece. If the test piece is twisted, one edge will be raised up off the flat surface. The gap between the raised test piece end and the flat surface may be estimated by inserting thickness shims of various sizes. The test piece twist is $\sin^{-1}(\text{gap}/4)$. See Figure 3 b).

NOTE 3 Some fully-articulating fixtures may have limitations in the ability to accommodate twisted test pieces. Users should be aware of these limitations if they exist, and avoid testing pieces with excessive twists.

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

- a) as-fired;
- b) machined by a customary-machining procedure;
- c) machined by a component-matched procedure;
- d) machined by the basic procedure defined in ISO 14704:2000, clause 6.2.5.

Consult ISO 14704 for additional details for these four options.

NOTE Surface preparation of test pieces can introduce machining flaws (especially microcracks beneath the test piece 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 Chamfers

The long edges shall be uniformly chamfered at 45° to a size of $0,12 \text{ mm} \pm 0,03 \text{ mm}$ as shown in Figure 3. They can alternatively be rounded with a radius of $0,15 \text{ mm} \pm 0,05 \text{ mm}$. 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 long axis. The maximum chamfer size permitted in this International Standard will cause a 1 % error in flexure strength. 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 shall be corrected for the reduced second moment of inertia of the test piece cross section in accordance with Annex B. As-fired test pieces may have chamfers applied before firing.

6.2.3 Test piece storage

The test pieces shall be handled with care 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.

6.2.4 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 (e.g., a Weibull analysis) is made.

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

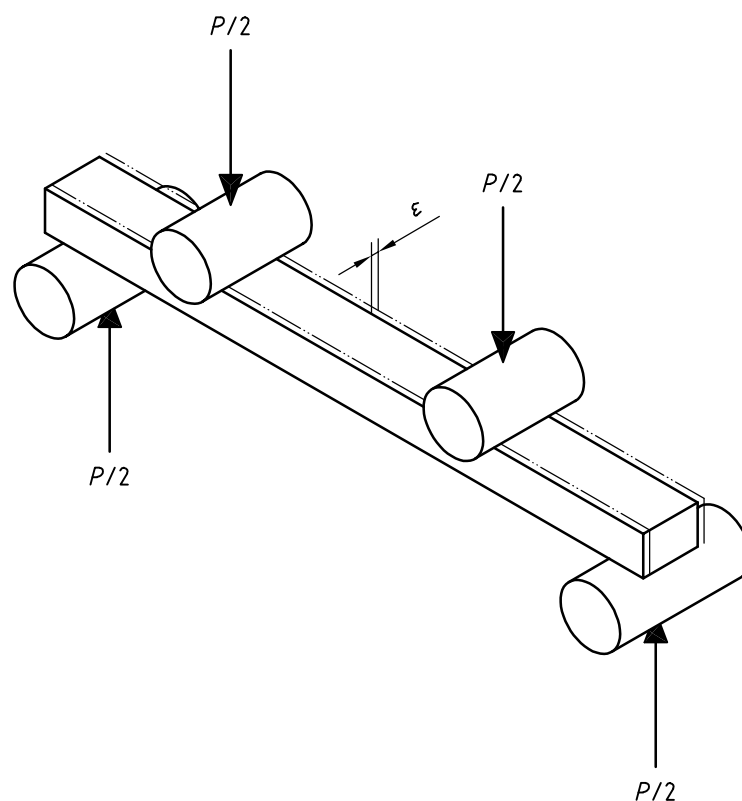
7 Procedure

7.1 Measure the test piece width, b , and height, h , 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 avoid introducing surface damage with the micrometer if measurements are made before the test.

7.2 Use an appropriate fixture in either the three- or four-point configuration. Four-point 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 cleaned of any fracture debris from previous tests, and that the bearings are cleaned of oxide scale or surface chemical reaction products. Ensure 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 (which is the side contacting the outer support span). Avoid damaging the test piece. Align the test piece carefully. Ensure that both test piece and fixture are centralized with respect to the loading axis, and that the test fixture rollers are in their correct starting positions. The test piece should have an approximately equal amount of overhang beyond the two outer bearings. Centre the test piece carefully so that the misalignment, ε , (Figure 4) is less than 0,1 mm of the axis of load application under the load application axis (front to back) as illustrated in Figure 4.



The misalignment, ε shall be less than 0,1 mm in accordance with 7.4.

Figure 4 — Alignment of the test piece under the axis of load application

NOTE 1 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.

NOTE 2 If the available furnace arrangements do not allow convenient access to the test jig for aligning the test piece, an alignment jig should be used. Alternatively, the alignment may be performed out of the furnace. It is essential that care be taken to ensure that the test piece does not move during this process. If there is a risk of movement, a temporary adhesive, such as polystyrene cement, may be used to fix the test piece and rollers in position. This burns off in the furnace, leaving all components correctly positioned.

7.5 Apply a slight preload, of no more than 10 % of the expected average fracture force, to the test piece whilst heating at 200 °C to 300 °C. 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.

NOTE Do not mark the test piece load points as is specified in the room temperature standard ISO 14704 since a marking material may contaminate the test piece or furnace.

7.6 A soft ceramic insert may be placed underneath the test piece to prevent the test piece fragments from being damaged if they impact the fixture after the test piece has fractured. Insulating felts or ceramic papers may be suitable but they should not chemically react with the fixture or test piece at the test temperature. The ceramic insert shall not interfere with the test piece alignment, exert a force on the test piece or fixtures, or inhibit fixture articulation or bearing rolling motions.

NOTE This ceramic insert will prevent unnecessary secondary fractures, and will help preserve the primary fracture pieces for subsequent fractographic analysis.

7.7 The test pieces shall be heated to the test temperature. Care shall be taken to ensure that the thermal expansion of the furnace does not cause the test piece preload to exceed 10 % of the expected average breaking force.

7.8 When the test temperature is reached, the test piece shall be maintained at this temperature for sufficient time for the temperature to stabilize. The time allowed for this shall be stated in the test report. The temperature measured by the thermocouple or other measuring device shall not vary during the time of the testing by more than $\pm 2^\circ\text{C}$.

7.9 The testing machine crosshead rate shall be 0,5 mm/min provided that the time to fracture is within the range of 3 s to 30 s. If the time to failure of a test piece is outside this range, then faster or slower crosshead rates shall be used so that the time to failure is within 3 s to 30 s.

NOTE 1 This crosshead rate will strain the test piece at a rate of approximately $1,0 \times 10^{-4}\text{s}^{-1}$.

NOTE 2 The effects of time-dependent phenomena, such as stress corrosion or slow crack growth on the measured flexural strength can be important for some sensitive materials, even for the relatively short times in the test.

7.10 Apply the test force at the specified rate and record the peak load at fracture. Measure the peak load with to accuracy of $\pm 1\%$ or better.

7.11 After completion of a test or a sequence of tests, retrieve the fracture fragments for later examination.

NOTE Only some pieces need be saved. Tiny fragments or tiny shards are often inconsequential since they do not contain the fracture origin. With some experience, it is usually not difficult to determine which pieces are important and should be retained. See ISO 14704:2000 Annex C for additional 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.

7.12 During a test series, observe the approximate fracture initiation location with the primary purpose of observing if fractures occurred within the inner span in four-point flexure. The bearing contact locations may often be detected on the test piece surface. Test piece fractures may occasionally occur outside the inner span or at an inner loading bearing. Test pieces with such fractures shall be included in the data set.

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.

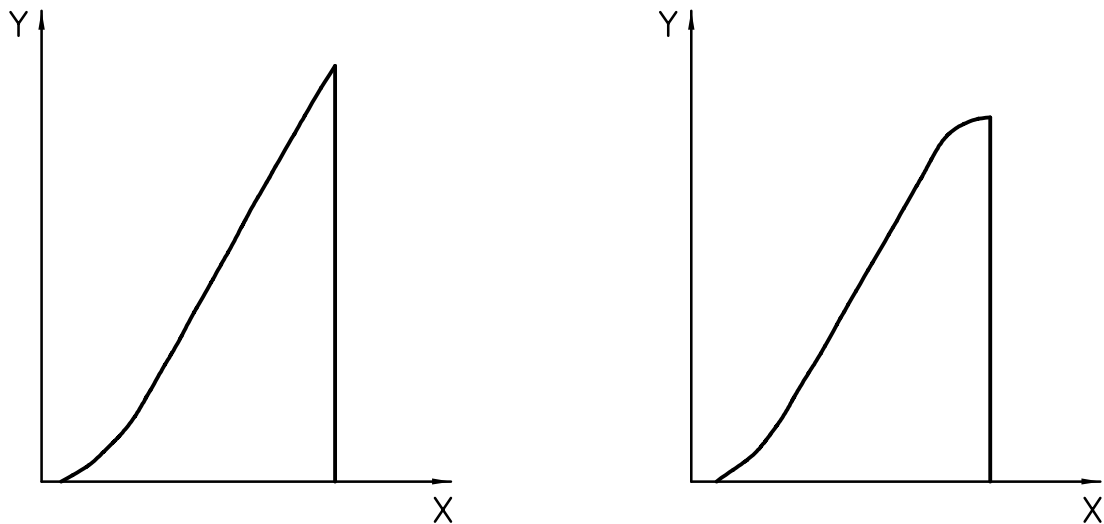
If many test pieces fracture from outside the inner span, or if many fractures begin directly under the inner load bearings in four-point flexure, there may be a fixture misalignment. Testing should stop until the problem is remedied. If the Weibull modulus is high, the number of fractures directly below the inner rollers will be increased due to the contact stresses which also influence the tensile side.

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 to be found in ISO 14704.

7.13 Inspect the bearings for signs of oxidation and/or reaction with the test piece or flattening, which would impair the performance of the jig. Renovate, clean or replace the bearings as necessary especially if they do not conform to the requirements of free rolling movement.

7.14 Inspect the force versus time (or displacement) record to determine whether there is any nonlinearity just before fracture as shown in Figure 5 b). Any nonlinearity just before fracture may indicate inelastic behaviour, and the assumptions of Clause 4 may be invalid. Report any evidence of inelastic behaviour.

NOTE Non-linear behaviour during initial loading at the low load portion of the record as shown in Figure 5 a) may simply indicate fixture settling or initial loading effects and is probably inconsequential.



a) Indicates linearly elastic behaviour

b) Indicates inelastic test piece behaviour, or possibly test fixture deformation or slippage

X Time (or displacement)

Y Force

Figure 5 — Force versus time (or displacement)

7.15 Measure and record the laboratory ambient relative humidity during the test sequence, unless the testing is in vacuum or inert atmosphere.

8 Calculation

8.1 Standard formula for the flexural strength in four-point flexure

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

$$\sigma_f = \frac{3Pa}{bh^2} \quad (1)$$

where

σ_f is the flexural strength in megapascals;

P is the fracture force in newtons;

a is the fixture moment arm, nominally 10,0 mm;

b is the test piece width in millimetres;

h is the test piece height, parallel to the direction of test force in millimetres.

NOTE The fixture moment arm, a , is nominally 10,0 mm for both fixtures specified by this standard. $a = L/4$ for 1/4-point, four-point flexure, and $a = L/3$ for 1/3-point, four-point flexure. The moment arm is also equal to $(L_2 - L_1)/2$ where L_2 and L_1 are the outer and inner spans, respectively. See Figure 1 and NOTE 2 in 8.2 below.

8.2 Standard formula for the flexural strength in three-point flexure

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

$$\sigma_f = \frac{3PL}{2bh^2} \quad (2)$$

where

σ_f is the flexural strength in megapascals;

P is the fracture force in newtons;

L is the fixture outer span in millimetres;

b is the test piece width in millimetres;

h is the test piece height, parallel to the direction of test force in millimetres.

NOTE 1 The three-point fixture outer span is either 40,0 mm or 30,0 mm as specified in this International Standard.

NOTE 2 Equations (1) and (2) are the customary and correct formulae for reporting the nominal flexural strength of a test piece. They give the maximum stress which occurred 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, e.g. 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 Corrections for chamfer size

If the chamfer sizes are larger than those specified in 6.2.2 and Figure 3, then the flexural strengths should be corrected as described in Annex B.

8.4 Corrections for thermal expansion

The thermal expansion of the test piece and the test fixtures will alter the physical dimensions of the fixture and test pieces. This will introduce a bias error of 1 % to 3 % in flexural strength if Equations (1) and (2) are used. Corrections for this expansion are listed in Annex C and may be made if agreed between the parties concerned. If the corrections are made, then this should be stated in the report.

8.5 Mean strength and standard deviation

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

$$\bar{\sigma}_f = \frac{\sum_1^n \sigma_{f,i}}{n} \quad (3)$$

$$s = \left[\frac{\sum_1^n (\sigma_{f,i} - \bar{\sigma}_f)^2}{n - 1} \right]^{1/2} \quad (4)$$

where

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

n is the total number of test pieces.

9 Accuracy and precision

9.1 The error in flexural strength for an individual test piece is estimated to be less than 3 % to 5 % if all the conditions of this test method are met.

NOTE Most tolerances have been chosen so that individual errors are typically 0,5 % or less. The chamfer error may be as large as 1 %, however.

9.2 As-fired test pieces may (or may not) have larger error, depending upon the conditions of the surface (flatness, evenness), uniformity of the test piece dimensions, uniformity of the cross section shape, and any twist or bow in the test piece.

10 Test report

Test report shall include the following information:

- a) nominal test temperature;
- b) furnace environment: air, vacuum, or inert atmosphere;
- c) type of heating element;
- d) temperature measuring device;
- e) mode of loading test pieces into the furnace (hot or cold);
- f) approximate rate of heating and the soak (or hold) time at temperature prior to the test piece test;
- g) 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;
- h) number of test pieces tested;
- i) if available, all relevant material data including vintage, billet or component identification number, and the date the material was manufactured;
- j) test piece preparation procedures, including all details of machining preparation; surface roughness (in both the longitudinal and transverse directions) on the test piece tensile surface if available;
- k) heat treatments or exposures, if any;
- l) flexural test environment, including humidity and temperature;
- m) crosshead rate in millimetres per minute and the approximate average time to fracture in seconds;

- n) for each test piece tested, $\sigma_{f,i}$, the flexural strength to three significant figures (e.g. 537 MPa);
- o) mean strength, σ_f , and the standard deviation, s , the following notation shall be used to report the mean strengths:
 - $\sigma_{(N,L)}$ to denote strengths measured in ($N = 4$ or 3)-point flexure, and ($L = 40$ mm or 30 mm) fixture outer span size

EXAMPLE 1

- $\sigma_{(4,40)} = 537$ MPa denotes the mean flexural strength was 537 MPa when measured in four-point flexure with 40 mm span fixtures;
- $\sigma_{(3,30)} = 580$ MPa denotes the mean flexural strength was 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:

$$\sigma_{(N,L)} = \text{XXX} [\text{RH \% or environment}]$$

to denote strengths measured in an atmosphere with RH, % relative humidity, or other environment

EXAMPLE 2

- $\sigma_{(4,40)} = 600$ MPa [45 %] denotes the mean flexural strength was 600 MPa when measured in four-point flexure with 40 mm span fixtures in laboratory ambient conditions with 45 % relative humidity;
- $\sigma_{(3,30)} = 575$ MPa [dry N₂] denotes the mean flexural strength was 575 MPa when measured in three-point flexure with 30 mm span fixtures in a dry nitrogen gas environment;
- $\sigma_{(3,30)} = 520$ MPa [vacuum] denotes the mean flexural strength was 520 MPa when measured in three-point flexure with 30 mm span fixtures in a vacuum environment.

- p) statement reporting whether flexural strengths have been corrected for thermal expansion;
- q) statement reporting whether the chamfers are within specifications; if they are not, then include a statement whether flexure strength has been corrected for the chamfer size;
- r) Any relevant comments concerning deterioration or sticking of the test jig, condition of as-fired or oxidized test pieces, the surface condition of the test pieces, the condition of the fracture surfaces, etc.;
- s) any indications of inelastic behaviour;
- t) any deviation(s) from the procedures described in this test method, and the reason for the deviation(s);
- u) name of testing laboratory, date of test, name of person conducting the tests, name of testing machine.

11 Strength scaling factors

The different test piece and fixture sizes permitted by this International Standard may produce different mean strengths. Annex D has 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 is to be used for design.

Flexural strength also may be influenced by many parameters associated with the test procedure. These include test environment, test piece size, test fixture details and test piece preparation. Flexure strength at elevated temperature may be strongly dependent upon loading rate, a consequence of creep, stress corrosion or slow crack growth. This test method is intended to measure flexure strength at fast loading rates in order to minimize these effects. If creep is active at the test temperature, stress relaxation may occur and the elastic formulation that is used to compute the strength will be in error.

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. See ISO 14704:2000 Annex A for additional information.

The specifications in this International Standard were chosen to provide a balance between controlling experimental error and 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 3 % to 5 %.

The fixture and corresponding test piece sizes in this International Standard are the same as the room temperature standard ISO 14704. The larger fixture-test piece (3 mm × 4 mm × 45 + mm) set exposes more material to the full stress, and produces a lower strength than the shorter fixture-test piece (3 mm × 4 mm × 35 + mm) set. This is normal for ceramics wherein fracture origins of different size or severity are distributed throughout the volume or surface. Weibull statistics often can correlate the strengths; e.g., 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. Annex D presents other conversion factors.

The three-point test configuration exposes only a very small portion of the test piece to maximum stress. Therefore, strengths are likely to be much greater than four-point flexural strength. Three-point flexure has some advantages. It uses simpler test fixtures, has less experimental error, 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^[5]. For additional information on experimental errors in flexural testing of ceramics, consult reference^[6].

Annex B (normative)

Chamfer correction factors

Flexural strengths shall 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, $I = (bh^3)/12$. For a rectangular cross section with four chamfered edges of size c , the adjusted moment of inertia from reference^[6] is:

$$I = \frac{bh^3}{12} - \frac{c^2}{9} \left(c^2 + \frac{1}{2} (3h - 2c)^2 \right) \quad (\text{B.1})$$

where the second term on the right hand side shows the reduction due to the chamfers. (A similar equation for rounded edges is available in Reference^[6].)

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 shall 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 5 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 the cross section is a simple rectangle) by a correction factor, F .

$$\sigma_f = F \sigma'_f \quad (\text{B.2})$$

Correction factors, F , for four chamfers or rounded edges are listed below.

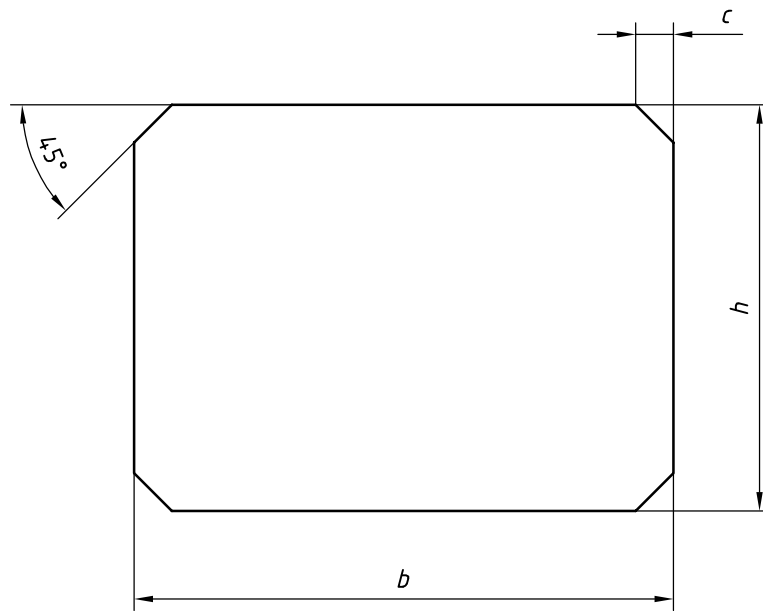


Figure B.1 — Chamfer geometry

Table B.1 — Correction factor, F , for four chamfers

c mm	Correction factor, F $b = 4 \text{ mm}, h = 3 \text{ mm}$
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
<hr/>	
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

NOTE The double line below $c = 0,150$ marks the limiting size for chamfers specified in 6.2.2 and Figure 3.

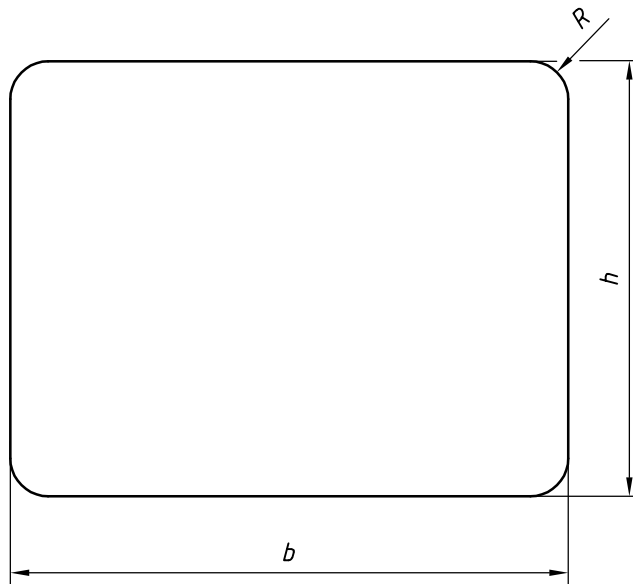


Figure B.2 — Rounded edge geometry

Table B.2 — Correction factor, F , for four rounded edges

R mm	Correction factor, F $b = 4 \text{ mm}, h = 3 \text{ mm}$
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
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

NOTE The double line below $R = 0,200$ marks the limiting size for chamfers specified in 6.2.2 and Figure 3.

Annex C (normative)

Corrections for thermal expansion

C.1 The test piece and the fixtures will expand with a rise in temperature. Depending upon the fixtures and test piece materials, the formula for strength may be in error by 0,5 % to several percent. The following equations may be used as alternatives to Equations (1) and (2) if the thermal expansion of the fixtures and test piece are known.

C.2 The corrected formula for the flexural strength in *four-point* flexure is:

$$\sigma_f = \frac{3Pa}{bh^2} \frac{(1,0 + \alpha_{\text{fix}}\Delta T)}{(1,0 + \alpha_{\text{spec}}\Delta T)^3} \quad (\text{C.1})$$

where

- σ_f is the flexural strength in megapascals;
- P is the fracture force in newtons;
- a is the fixture moment arm, nominally 10,0 mm;
- b is the test piece width in millimetres;
- h is the test piece height, parallel to the direction of test force in millimetres;
- α_{fix} is the average coefficient of thermal expansion from room temperature to the test temperature for the test fixture material per degree centigrade;
- α_{spec} is the average coefficient of thermal expansion from room temperature to the test temperature for the test piece material per degree centigrade;
- ΔT is the temperature difference from room to test temperature per degree centigrade.

NOTE a is 10,0 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.)

C.3 The corrected formula for the flexural strength in *three-point* flexure is:

$$\sigma_f = \frac{3PL}{2bh^2} \frac{(1,0 + \alpha_{\text{fix}}\Delta T)}{(1,0 + \alpha_{\text{spec}}\Delta T)^3} \quad (\text{C.2})$$

where

- σ_f is the flexural strength in megapascals;
- P is the fracture force in newtons;
- L is the fixture outer span in millimetres;
- b is the test piece width in millimetres;
- h is the test piece height, parallel to the direction of test force in millimetres;

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α_{fix} is the average coefficient of thermal expansion from room temperature to the test temperature for the test fixture material per degree centigrade;

α_{spec} is the average coefficient of thermal expansion from room temperature to the test temperature for the test piece material per degree centigrade;

ΔT is the temperature difference from room to test temperature per degree centigrade.

NOTE The three-point fixture outer span is either 40,0 mm or 30,0 mm as specified in this International Standard.

Annex D (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. 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:

$$\frac{\sigma_1}{\sigma_2} = \left(\frac{V_{E2}}{V_{E1}} \right)^{\frac{1}{m}} \tag{D.1}$$

where

σ_1 is the mean strength of test piece/fixture type 1;

σ_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:

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

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

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;

$$\frac{\sigma_{4,30}}{\sigma_{4,40}} = \left(\frac{V_{E,4,40}}{V_{E,4,30}} \right)^{\frac{1}{m}} = W \tag{D.4}$$

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

Table D.1 — W versus Weibull modulus, m , for Weibull volume or surface scaling

	Weibull modulus, 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 distributions, then:

$$\frac{\sigma_1}{\sigma_2} = \left(\frac{S_{E2}}{S_{E1}} \right)^{\frac{1}{m}} \tag{D.5}$$

where

σ_1 is the mean strength of test piece/fixture type 1;

σ_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:

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

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

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

$$\frac{\sigma_{4,30}}{\sigma_{4,40}} = \left(\frac{S_{E,4,40}}{S_{E,4,30}} \right)^{\frac{1}{m}} = W \tag{D.8}$$

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

NOTE 1 This surprising outcome can be confirmed by algebraic manipulation of the above equations. 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.

Annex E (informative)

VAMAS round robin

A round robin project on elevated temperature flexure strength was conducted under the auspices of the Versailles Advanced Materials and Standards (VAMAS) program in 1999-2000. Thirteen laboratories in six countries measured the strength of silicon nitride at 1 200 °C in air. Semi- and fully-articulating fixtures were used. All testing was in four-point flexure, with either 10 mm × 30 mm or 20 mm × 40 mm spans. Most laboratories tested 10 or 12 specimens [7].

Some of the conclusions from this project are as follows.

- a) The strengths of specimens tested with the 10 mm × 30 mm spans were slightly greater (6,3 %) than the strengths of specimens tested with 20 mm × 40 mm spans. The difference in average strengths was primarily due to the difference in Weibull effective volumes or effective areas. (The Weibull modulus was approximately 10.)
- b) Specimens tested on fully-articulated fixtures were slightly stronger (5,1 %) than specimens tested on semi-articulated fixtures.
- c) The limited number of specimens tested by each laboratory led to a large reproducibility uncertainty (between-laboratory strength variations). Much of the difference can be attributed to small sample size statistical effects and the differences are within the confidence intervals predicted by Weibull statistics.
- d) Supplemental experiments confirmed that friction constraints may affect load-displacement curve data (and presumably the measured flexure strength) with fixtures having rollers that are not completely free to roll. Fixtures with rollers in square slots of insufficient clearance may inhibit roller motion.
- e) Additional testing in an inert nitrogen environment was performed by two laboratories. Nitrogen-tested specimens were weaker than air-tested specimens, presumably due to oxidative crack healing in the latter.
- f) Load-displacement curves were valuable in interpreting the performance of the test fixtures and for confirming that the material has linearly-elastic behaviour up to fracture.

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