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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for flexural bond strength of ceramics

Céramiques techniques (céramiques avancées, céramiques techniques avancées) – Méthodes d'essai pour déterminer la résistance d'adhésion en flexion des céramiques

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Foreword

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

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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for flexural bond strength of ceramics

1 Scope

This document specifies a test method for determining the flexural bond strength of ceramic/ceramic joints or ceramic/metal joints at room temperature. The substrate materials, for example ceramic or metal, are both monolithic. This method can be used to test the interfacial bond strength of the joint under bending conditions. It can be used for the development of joining materials and/or for the quality control of joints, the characterization and generating design data purposes.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3611, *Geometrical product specifications (GPS) — Dimensional measuring equipment: Micrometers for external measurements — Design and metrological characteristics*

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

ISO 14704:2016, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for flexural strength of monolithic ceramics at room temperature*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
flexural bond strength
maximum stress at fracture at the bond interface of a specified beam containing the ceramic/ceramic joint or the ceramic/metal joint subjected to bending

3.2
four-point flexure
configuration of flexural test where a specimen is subjected to equal loads through two bearings symmetrically located between two support bearings

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

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

3.3
four-point 1/4 point flexure
specific configuration of four-point flexural test where the inner bearings are situated one-quarter of the support span away from the two outer bearings

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

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

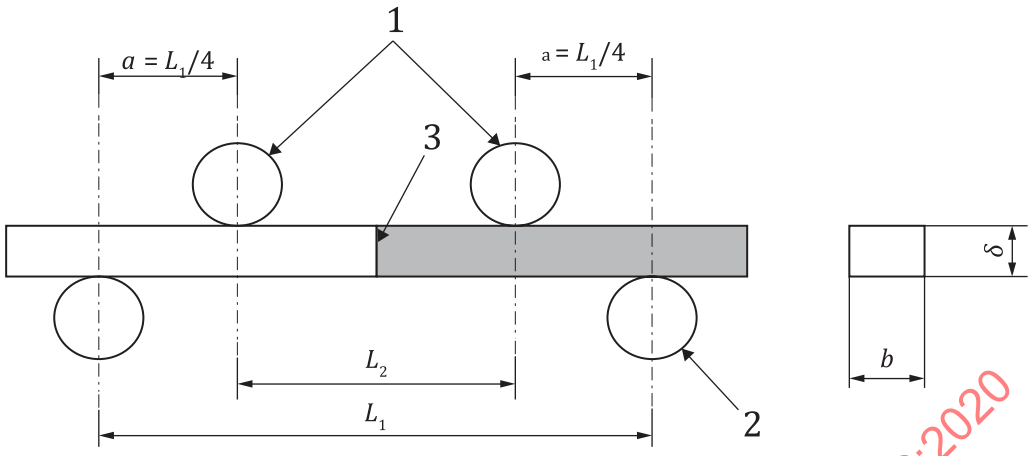
Note 1 to entry: See Figure 1 b) and Table 1.

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

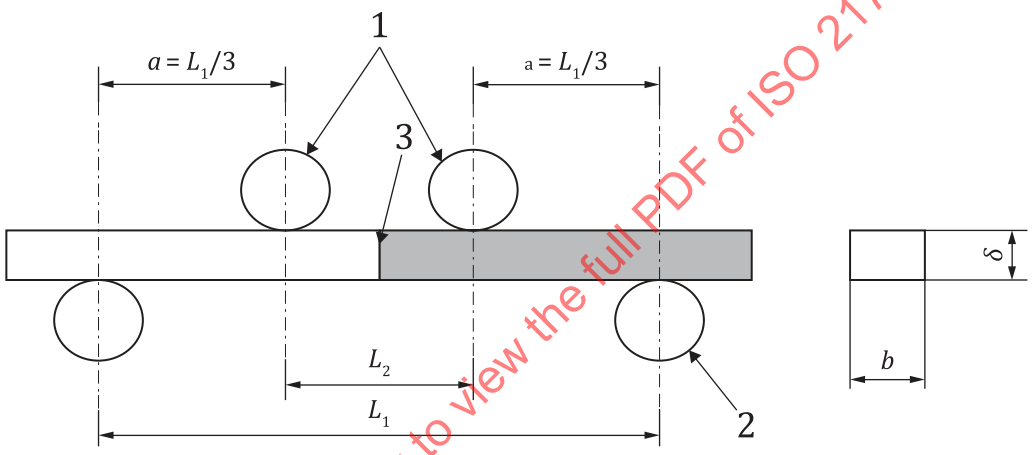
3.6
fully articulating fixture
test fixture designed to apply uniform and even loading to specimens that may have uneven, non-parallel or twisted surfaces

4 Principle

A beam-type specimen containing the specific bond joint with a rectangular cross-section or circular cross-section is loaded in flexure until fracture. The load at fracture, the geometry of the test fixture and specimen dimensions are used to compute the flexural bond strength, which is a measure of the bond strength. The material or joining interface is assumed to be linearly elastic.



a) Four-point 1/4 point flexure



b) Four-point 1/3 point flexure

Key

1	loading bearings	b	width
2	support bearings	δ	thickness
3	interface	L_1	outer span
a	distance between loading and support bearings	L_2	inner span

Figure 1 — Flexural bond test configurations

Table 1 — Dimensions of flexural bond test configurations

Dimensions in millimetres

Bending type	Specimen	L_1 (outer span)	L_2 (inner span)	a	Diameters of bearings
Four-point 1/4	A-I, B-I	40 ± 0,1	20 ± 0,1	10 ± 0,1	4,5 to 5,0
Four-point 1/3	A-II, B-II	30 ± 0,1	10 ± 0,1	10 ± 0,1	4,5 to 5,0
Four-point 1/3	A-III, A-IV, B-III, B-IV	30 ± 0,1	10 ± 0,1	10 ± 0,1	4,0 to 6,0
Four-point 1/3	A-V, A-VI, B-V, B-VI	(4,5~10,5)δ ± 0,5	(1,5~3,5)δ ± 0,5	(1,5~3,5)δ ± 0,5	4,0 to 6,0

5 Apparatus

5.1 Testing machine

A suitable testing machine capable of applying a uniform crosshead speed shall be used. The testing machine shall have the function for recording the peak load applied to the test specimen. The testing machine shall fulfil the requirements of ISO 7500-1, Class 1, with an accuracy of 1 % of the indicated load at fracture.

5.2 Test fixture

5.2.1 General

The test adopts four-point flexure configurations, 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 it can articulate or tilt to ensure uniform loading to the specimen. The articulation is designed so that bearing parts of the fixture can rotate, as shown in ISO 14704:2016, Figure B.1. The bearing parts should also provide articulation to ensure that all bearing contacts can apply uniform load to the specimen surfaces. Semi-articulated fixtures, which have pairs of upper and lower bearings articulating to match the specimen surfaces, may be used with specimens that have flat and parallel surfaces, as illustrated in ISO 14704:2016, Figures B.2 a) and B.3 a). Fully articulated fixtures have more moving parts and are necessary for specimens that do not have flat and parallel surfaces, as illustrated in ISO 14704:2016, Figures B.2 b) and B.3 b).

5.2.2 Bearings

The loading and supporting bearings of specimens may be cylindrical rollers or cylindrical bearings. The specifications for bearings shall be as given in ISO 14704.

5.2.3 Positioning of bearings

The requirements for the positioning of bearings shall be as given in ISO 14704. In addition, the joining interfaces should be positioned at the centre of the inner bearings to within ± 0,1 mm.

5.2.4 Fixture material

The fixture should not be permanently deformed by bearings. The requirements and recommendations for fixture materials shall be as given in ISO 14704.

5.3 Micrometer

A micrometer as described in ISO 3611 but with a resolution of 0,002 mm, shall be used to measure the specimen dimensions (see ISO 14704). 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 Type A specimens

Type A specimens have one interface. Test specimen dimensions are shown in Figure 2 and Table 2. Cross-sectional tolerance shall be ±0,2 mm. The parallelism tolerance on opposite longitudinal faces is 0,015 mm. It is recommended that the interface be located near the centre of the test specimen. The interfaces shall be perpendicular to the longitudinal direction of the test specimen. It is recommended

that the A-I, A-II, A-III and A-IV type specimens be employed. If those standard-sized specimens are difficult to prepare due to constraints of the joining method or joining configuration, specimens with other dimensions (A-V and A-VI types, rectangular or circlular cross-section) are permitted by mutual agreement between the customer and the vendor. In this case, the total length of the specimen, L_T, shall be six times larger than the width (or diameter) of the specimen and both right and left lengths, L_A and L_B, shall be three times larger than the width (or diameter) of the specimen.

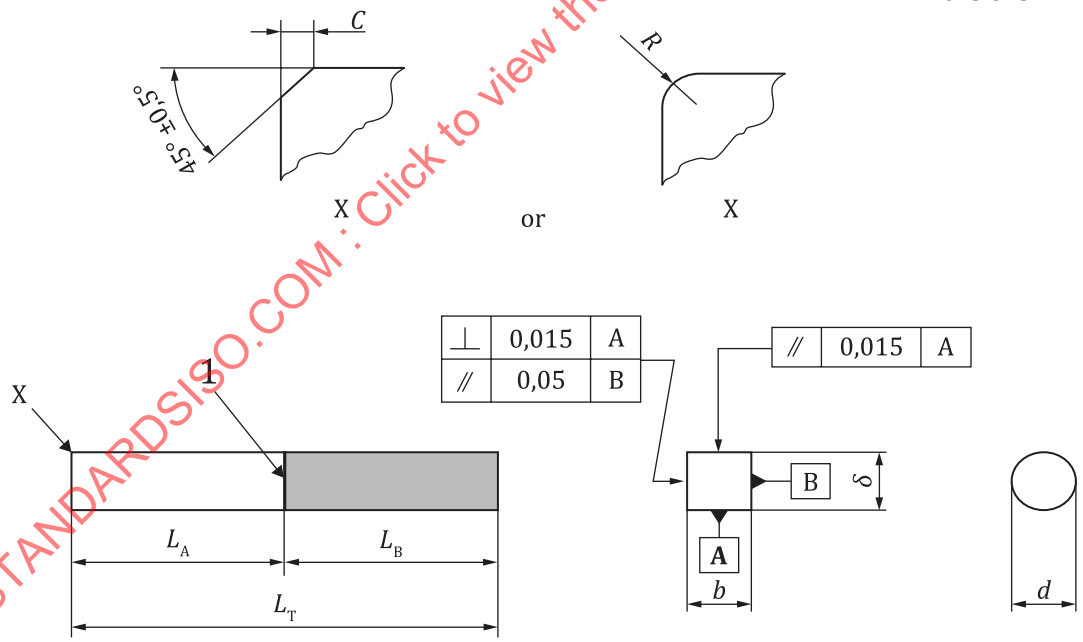
6.1.2 Type B specimens

Type B specimens have two or more interfaces. Test specimen dimensions are shown in Figure 3 and Table 3. The tolerances are the same as Type A. The interface shall be at the centre of the test specimen and shall be perpendicular to the longitudinal direction of the test specimen. Specimen types B-I, B-II, B-III and B-IV are preferred. If these standard-sized specimens are difficult to prepare due to constraints of the joining method or joining configuration, specimens with other dimensions (B-V and B-VI types, rectangular or circular cross-section) are permitted by mutual agreement between the customer and the vendor. In this case, the total length of the specimen, L_T, shall be six times larger than the width (or diameter) of the specimen and both right and left lengths, L_A and L_B, shall be three times larger than the width (or diameter) of the specimen. In addition, the middle length, L_C, shall be b (or d) unless there is geometrical constraint due to a joining condition or a joining configuration.

6.1.3 Chamfering and rounded edge

All four long edges shall be chamfered or rounded as shown in Figures 2 and 3. The dimensions of chamfering, C, and rounded edge, R, are shown in Table 4.

Dimensions in millimetres



Key

1	interface of the ceramic joint	d	diameter
C	chamfering	L_T	total length
R	rounded edge	L_A	left length
b	width	L_B	right length
δ	thickness		

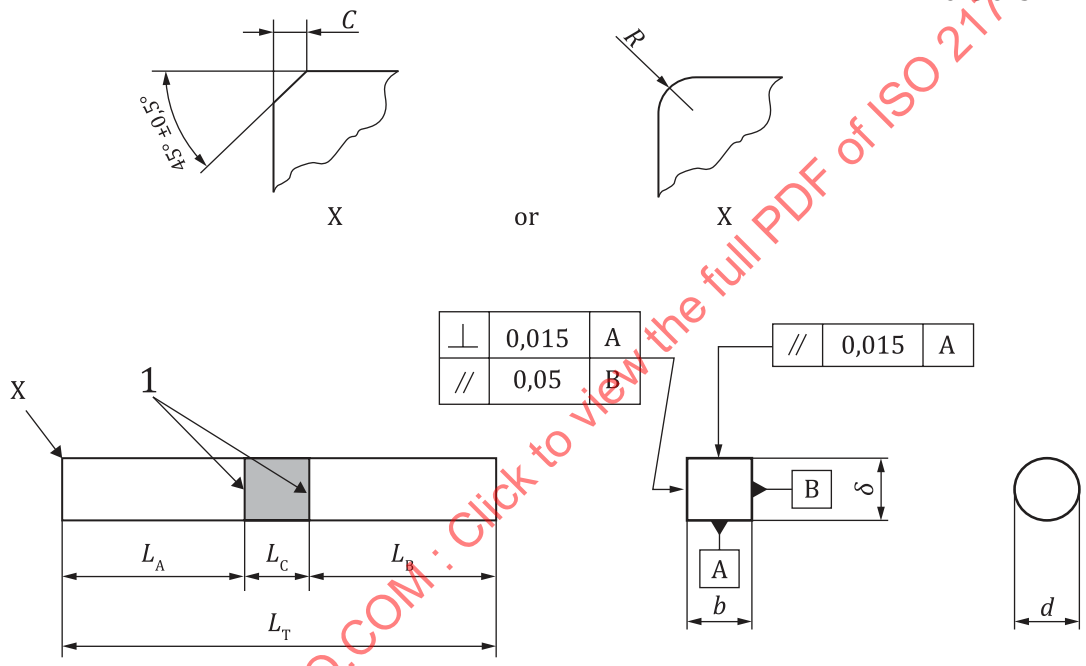
Figure 2 — Type A specimen

Table 2 — Dimensions of Type A specimen

Dimensions in millimetres

Specimen	Cross-sectional shape	Width	Thickness	Diameter	Total length	Left length	Right length
A-I	Rectangular	4 ± 0,2	3 ± 0,2	—	≧45	≧22,5	≧22,5
A-II	Rectangular	4 ± 0,2	3 ± 0,2	—	≧35	≧17,5	≧17,5
A-III	Rectangular	6 ± 0,2	6 ± 0,2	—	≧35	≧17,5	≧17,5
A-IV	Circular	—	—	ϕ6 ± 0,2	≧35	≧17,5	≧17,5
A-V	Rectangular	b ± 0,2	δ ± 0,2	—	≧6b	≧3b	≧3b
A-VI	Circular	—	—	d ± 0,2	≧6d	≧3d	≧3d

Dimensions in millimetres



Key

1 interface of the ceramic joint
C chamfering
R rounded edge
b width
δ thickness
d diameter
L_T total length
L_A left length
L_B right length
L_C middle length

Figure 3 — Type B specimen

Table 3 — Dimensions of Type B specimen

Dimensions in millimetres

Specimen	Cross-sectional shape	Width	Thickness	Diameter	Total length	Left length	Middle length[a]	Right length
B-I	Rectangular	4 ± 0,2	3 ± 0,2	—	≧45	≧22,5	4	≧22,5
[a] If there is a geometrical constraint due to a joining condition or a joining configuration, other values are permitted.								

Table 3 *(continued)*

Specimen	Cross-sectional shape	Width	Thickness	Diameter	Total length	Left length	Middle length[a]	Right length
B-II	Rectangular	4 ± 0,2	3 ± 0,2	—	≧35	≧17,5	4	≧17,5
B-III	Rectangular	6 ± 0,2	6 ± 0,2	—	≧35	≧17,5	6	≧17,5
B-IV	Circular	—	—	6 ± 0,2	≧35	≧17,5	6	≧17,5
B-V	Rectangular	b ± 0,2	δ ± 0,2	—	≧6b	≧3b	b	≧3b
B-VI	Circular	—	—	d ± 0,2	≧6d	≧3d	d	≧3d

[a] If there is a geometrical constraint due to a joining condition or a joining configuration, other values are permitted.

Table 4 — Dimensions of chamfering and rounded edge

Dimensions in millimetres

Specimen	Cross-sectional shape	Chamfering	Rounded edge
AI, BI	Rectangular	0,12 ± 0,03	0,15 ± 0,05
AII, BII	Rectangular	0,12 ± 0,03	0,15 ± 0,05
AIII, BIII	Rectangular	0,2~0,4	0,2~0,4
AV, BV	Rectangular	(0,03~0,07)b	(0,03~0,07)b

6.2 Specimen preparation

6.2.1 General

This document allows different options for preparing specimens. There are two major production methods for the specimen. The first method is cutting from a larger ceramic jointed body followed by grinding and polishing. The other method is joining the pair of blocks which have a size which is a little larger than half of the standard-sized specimens described in Figures 2 and 3. The outer surfaces of the specimens when they have been joined together shall be ground and polished to obtain flat surfaces.

All four long edges shall be chamfered or rounded, as shown in Figures 2 and 3. In all cases, the end faces of the specimen do not need special preparation or finishing. Although a surface finish specification is optional, it is highly recommended that the surface roughness be measured and reported.

6.2.2 Custom machining procedure

In cases where a custom machining procedure has been developed to produce specimens satisfying the requirements, this custom 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 Figures 2 and 3.

6.2.3 Component-matched procedure

The specimen shall have the same surface preparation as that given to a component. The test report should include contents as specified in 6.2.2.

6.2.4 Basic machining procedure

6.2.4.1 If the procedures in 6.2.2 to 6.2.3 are not applicable, then the following procedure may be used.

NOTE The procedure specified below is a conservative practice. It is intended to minimize machining damage in a broad range of ceramics. Faster or more aggressive removal rates can be sought if needed.

6.2.4.2 The connection method of the junction surface should depend on the real connection situation of the components.

6.2.4.3 To ensure smoothness, the whole specimen should be ground integrally after combing two parts of material. Specimens shall be ground in the longitudinal direction, as shown in Figure 4.

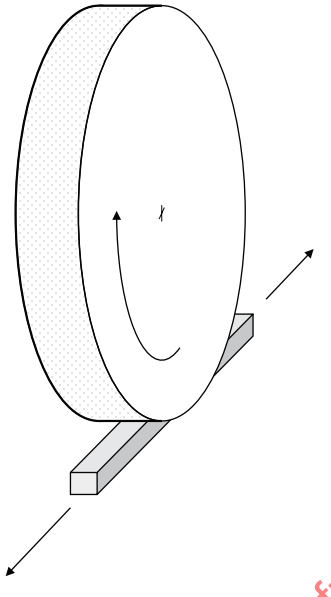


Figure 4 — Specimen surface grinding in the longitudinal axis

6.2.4.4 The grinding procedure shall be as given in ISO 14704:2016, 6.2.5.2 to 6.2.5.5. The final dimensions of the specimen shall be in accordance with 6.1.1 and 6.1.2 as well as Tables 2 and 3.

6.2.5 Parallelism, orthogonality and chamfer sizes

Details shall be as given in ISO 14704:2016, 6.2.6.

6.2.6 Handling of specimens

Details shall be as given in ISO 14704:2016, 6.2.7.

6.2.7 Number of specimens

A minimum of 10 specimens shall be required for the purpose of estimating the mean flexural strength.

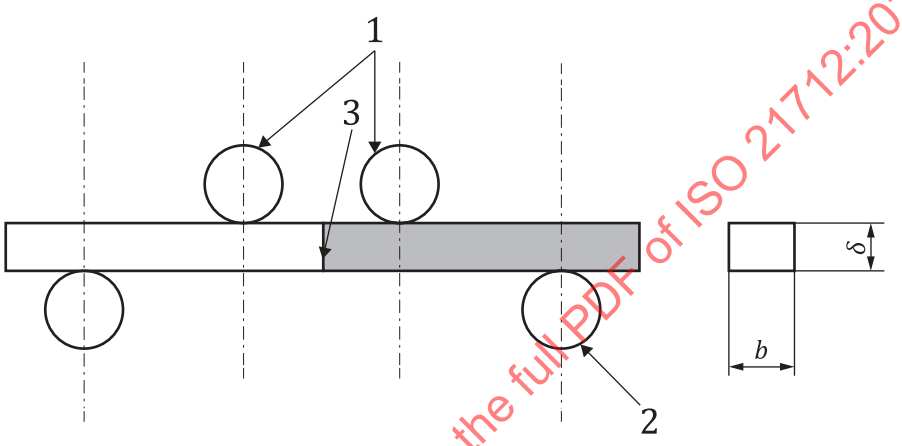
7 Procedure

7.1 Measure the specimen width, b, and thickness, δ, or radius, d, with a resolution of 0,002 mm. The specimen size may be measured either before or after the test. If the specimen is measured before the test, measure the specimen dimensions at the joint; otherwise, measure the specimen dimensions near the joint location after the test. Care shall be taken to not introduce surface damage when using the micrometer.

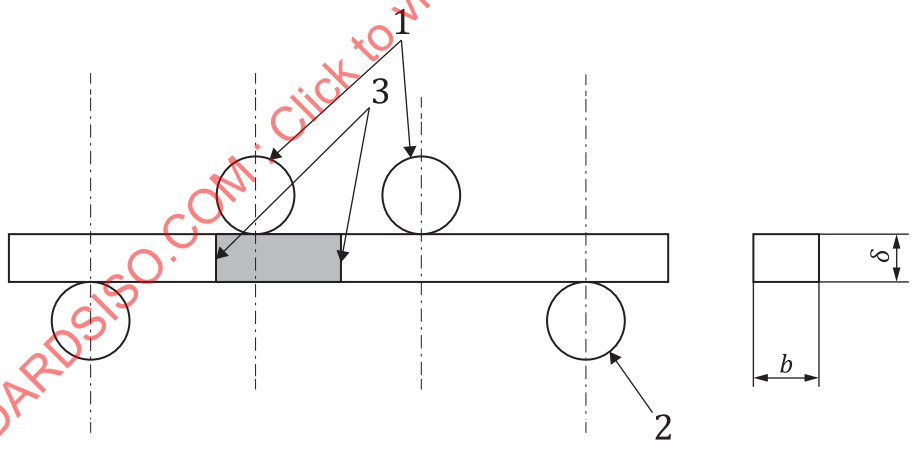
7.2 Test the specimens on the appropriate fixture in the four-point configuration. A fully articulating fixture shall be used if the specimen parallelism requirements cannot be met.

7.3 Ensure that the test fixtures are clean, that the bearings are free of burrs or deep scratches and that the bearings are free to roll and articulate.

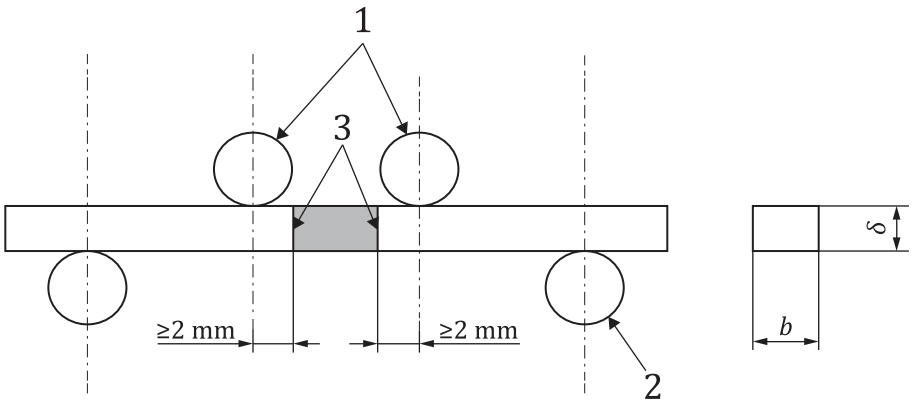
7.4 Place each specimen in the test fixture with the wider face resting on the bearings if the cross-section is rectangular. There are three types of relationship between the interface of the joints and bearings as shown in Figure 5. When one interface is investigated, the interface shall be located at the centre of the loading bearings [Figure 5 a) and b)]. When two interfaces are investigated simultaneously [Figure 5 c)], the two interfaces shall be symmetrical with the centre line of the fixture. The two interfaces shall be within the loading bearings. The contact points of loading bearings shall be located 2 mm or more away from each interface. The test results, pass or fail, for each interface shall be reported. Align the specimen carefully. The specimen should have an approximately equal amount of overhang beyond the two outer bearings. Centre the specimen carefully within 0,1 mm of the axis of load application (front to back), as illustrated in Figure 6. Positioning stops for the specimen are strongly recommended. This is especially important with fully articulating fixtures which may cause the specimen to shift during articulation. The fixture design should not allow excessive shifts.



a) Type A specimen



b) Type B specimen (one interface is examined)

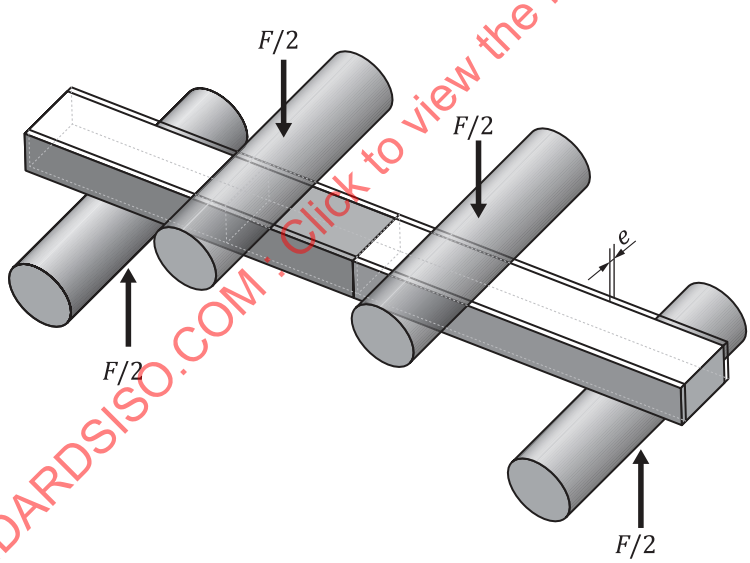


c) Type B specimen (two interfaces are examined simultaneously)

Key

1	loading bearings	*b*	width
2	support bearings	*δ*	thickness
3	interface		

Figure 5 — Positional relationship between interface and bearings



Key

e position tolerance of the specimen from the axis centre of load application (front to back), <0,1 mm

Figure 6 — Alignment of the specimen under the axis of load application

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

7.6 Gently mark the specimen to identify the approximate locations of the two inner loading bearings. Also mark the specimen so that the compression surface can be distinguished from the tensile surface.