
**Fine ceramics (advanced ceramics,
advanced technical ceramics) —
Mechanical properties of ceramic
composites at elevated temperature
in air atmospheric pressure —
Determination of in-plane shear
strength**

Céramiques techniques (céramiques avancées, céramiques techniques avancées) — Propriétés mécaniques des composites céramiques à température élevée sous air à pression atmosphérique — Détermination de la résistance au cisaillement dans le plan

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Contents

	Page
Foreword	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	2
5 Significance and use	5
5.1 Test environment.....	5
5.2 Material orientation.....	6
5.3 Preparation of test pieces.....	6
5.4 Failures outside gauge section.....	6
5.5 Thin test pieces.....	6
6 Apparatus	6
6.1 Test machine.....	6
6.2 Loading devices.....	6
6.3 Test fixture.....	6
6.4 Heating apparatus.....	7
6.5 Temperature measurement.....	7
6.6 Data recording system.....	7
6.7 Dimension-measuring devices.....	7
7 Test pieces	7
7.1 Test piece geometry.....	7
7.2 Test piece preparations.....	8
7.3 Number of specimen tests.....	8
8 Test conditions	8
8.1 Test modes and rates.....	8
8.1.1 General.....	8
8.1.2 Displacement rate.....	8
8.1.3 Load rate.....	8
8.2 Test temperature.....	8
9 Procedures	9
9.1 Test piece dimensions.....	9
9.2 Test preparation.....	9
9.3 Test implementation.....	9
9.3.1 Mounting of test piece on test fixture.....	9
9.3.2 Heating of test piece.....	9
9.3.3 Loadings.....	10
9.4 Completion of testing.....	10
9.5 Test validity.....	11
10 Calculation of results	11
10.1 Shear strength.....	11
10.2 Error calculation.....	11
11 Test report	12
Annex A (informative)	13
Bibliography	16

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Mechanical properties of ceramic composites at elevated temperature in air atmospheric pressure — Determination of in-plane shear strength

1 Scope

This document specifies a method for the determination of in-plane shear strength of continuous fibre-reinforced ceramic composites at elevated temperature in air or inert atmosphere by the asymmetric four-point bending test on double-edge notched specimens. The shear strength in plane (1,2) can be evaluated, where direction 1 is that of the greater fraction of reinforcement and direction 2 is perpendicular to direction 1. Methods for test piece fabrication, testing modes and rates (load or displacement rate), data collection and reporting procedures are addressed.

This document applies to all ceramic matrix composites with continuous fibre-reinforcement: unidirectional (1D), bidirectional (2D) and tridirectional (xD, with $2 < x \leq 3$).

This document is for material development, material comparison, quality assurance, characterization, reliability and design data generation.

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 19634, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Ceramic composites — Notations and symbols*

ISO 20507, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Vocabulary*

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

IEC 60584-2, *Thermocouples — Part 2: Tolerances*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 19634 and ISO 20507 and the following 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
initial gauge section

S_0
initial area of test piece between notch roots

3.2
test temperature

T
temperature measured at the centre of the gauge section

3.3
applied force

F
force applied to a test piece

3.4
shear failure force

F_{\max}
maximum force required to fracture a shear-loaded test piece

3.5
shear strength

τ_m
maximum shear stress which a material is capable of sustaining

Note 1 to entry: Shear strength is calculated from the shear failure force and the gauge section.

3.6
inner span

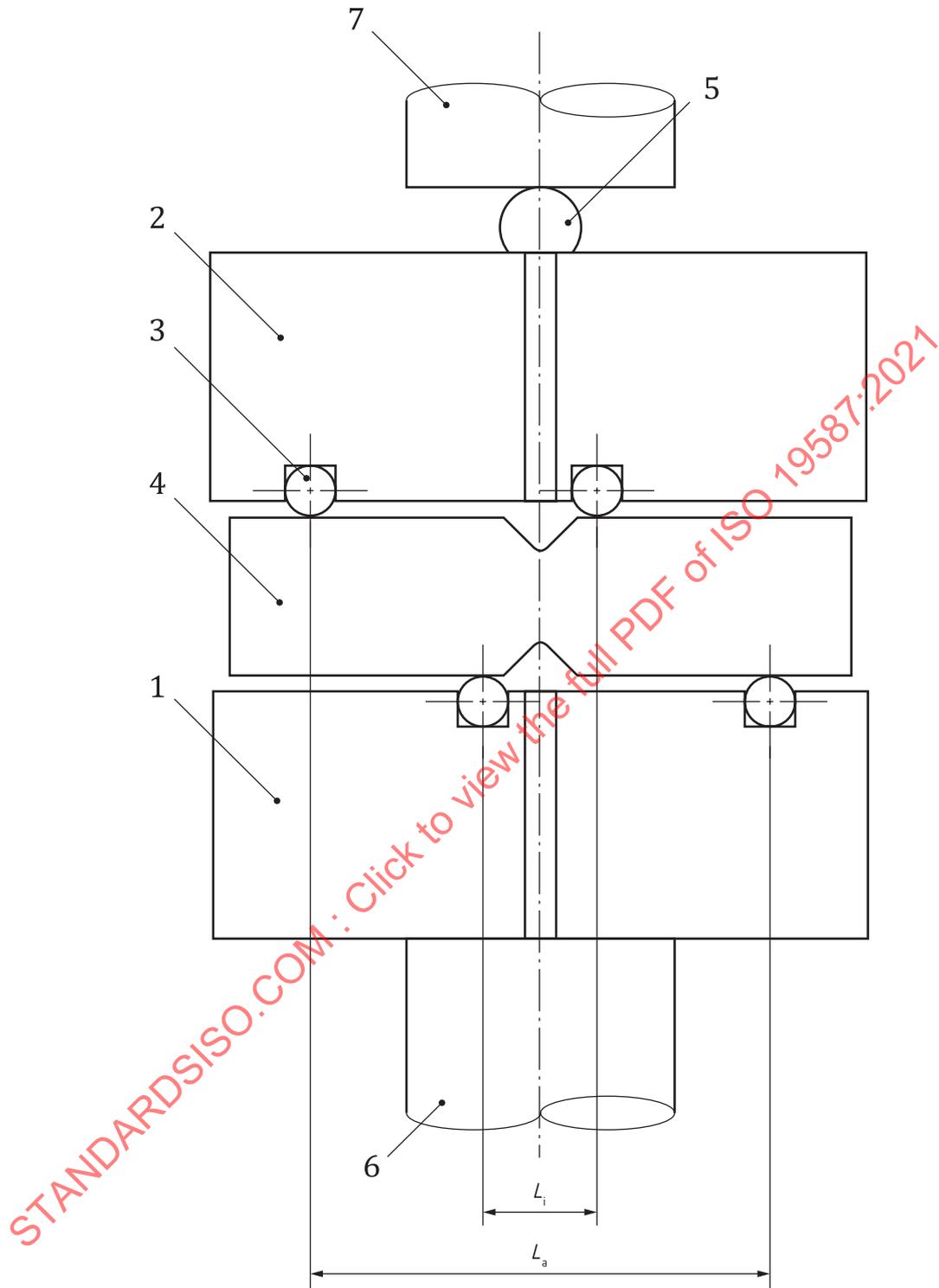
L_i
centre distance between two inner loading pins

3.7
outer span

L_a
centre distance between two outer loading pins

4 Principle

The in-plane shear strength of continuous fibre-reinforced ceramic composites, as determined by this document, is measured by the asymmetric four-point bending test at elevated temperature in air or inert atmosphere. According to this test, the shear strength is determined by loading a test coupon in the form of a rectangular flat strip with symmetric, centrally located V-notches using a mechanical testing machine and an asymmetric four-point bending fixture. Failure of the test piece occurs by shear force between the V-notches. Schematics of the test set-up and the test piece are shown in [Figures 1](#) and [2](#), respectively. The free body, bending moment and shear force diagrams by the asymmetric four-point bending flexure are illustrated in [Figure 3](#).



Key

- | | | | |
|---|---------------|---|---------------|
| 1 | lower fixture | 2 | upper fixture |
| 3 | loading pin | 4 | test piece |
| 5 | loading ball | 6 | lower ram |
| 7 | upper ram | | |

Figure 1 — Schematics of asymmetric four-point bending test set-up

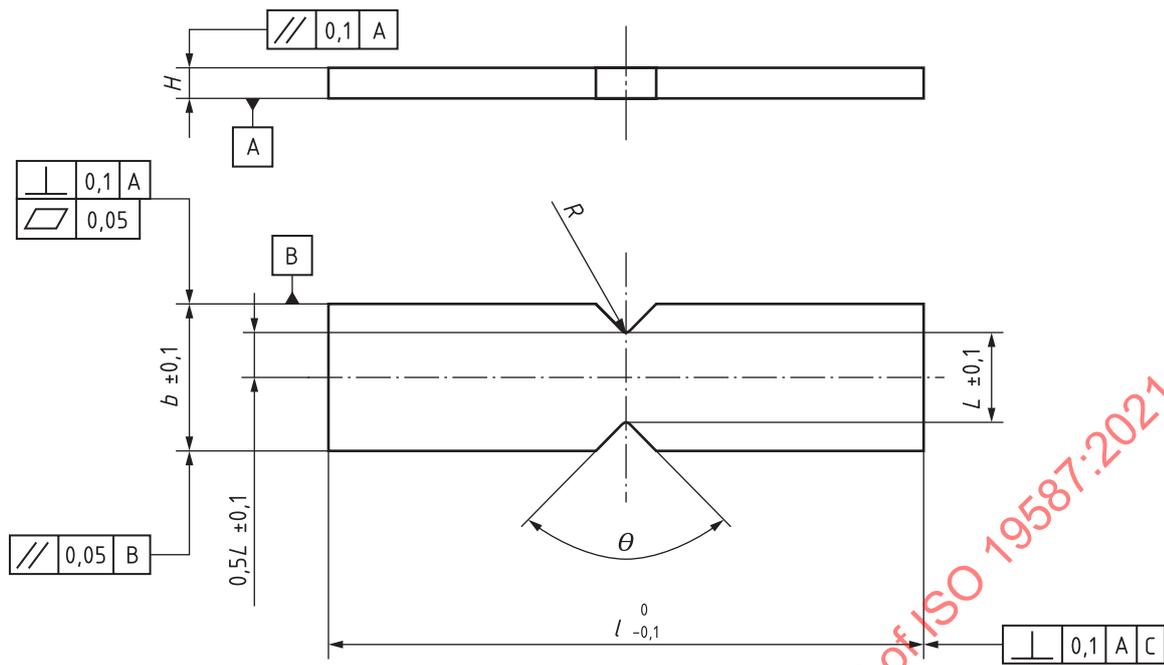
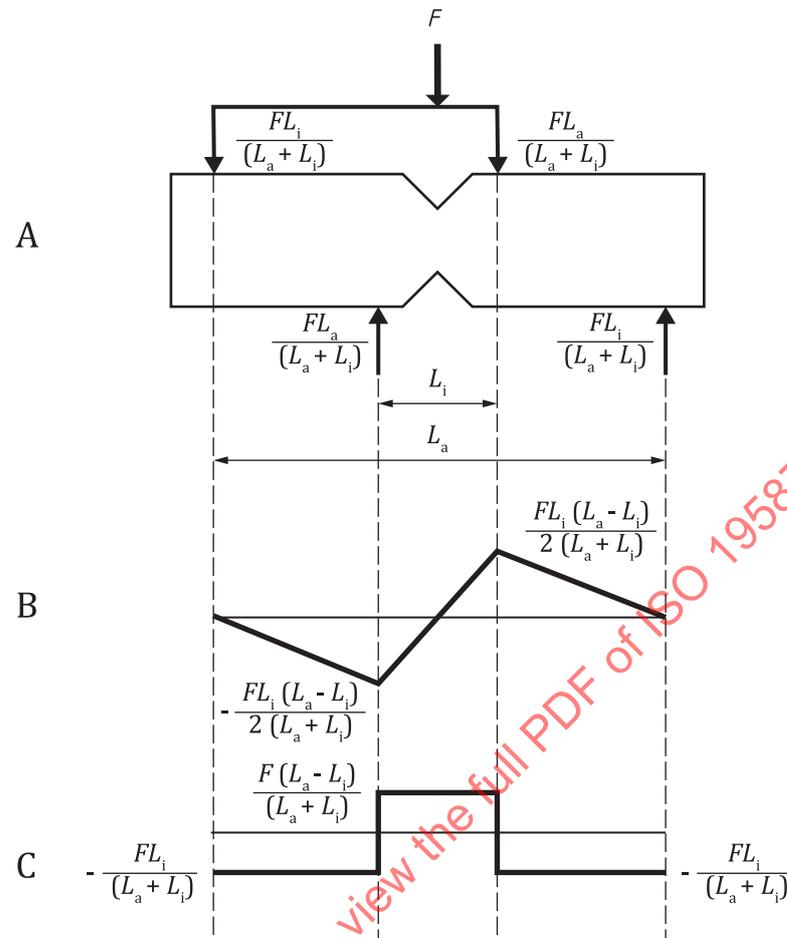


Figure 2 — Geometry and dimensions of test piece

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**Key**

- A free body diagram
- B bending moment diagram
- C shear force diagram

Figure 3 — Free body, bending moment and shear force diagrams of asymmetric four-point bending

5 Significance and use

5.1 Test environment

The test environment can have an influence on the measured shear strength. In particular, the behaviour of materials susceptible to slow-crack-growth fracture will be strongly influenced by the test environment and testing rate. Testing to evaluate the maximum strength potential of a material shall be conducted in inert environments, at sufficiently rapid testing rates or both, so as to minimize slow-crack-growth effects. Conversely, testing can be conducted in environments and testing modes and rates representative of service conditions to evaluate material performance under those conditions. When testing is conducted in uncontrolled ambient air with the objective of evaluating maximum strength potential, water partial pressure and temperature shall be monitored and reported, if the tested materials are sensitive to these parameters.

5.2 Material orientation

In this method, the shear strength in plane (1,2) can be evaluated, where direction 1 is that of the greater fraction of reinforcement and direction 2 is perpendicular to direction 1. Either direction 1 or direction 2 should be along the length of the test piece. The material properties in plane (1,3) or plane (2,3) can be interlinear properties for laminated materials. Thus, this method is targeted for evaluation of the shear strength in plane (1,2).

NOTE For the definition of material orientation, see ISO 19634.

5.3 Preparation of test pieces

Preparation of test pieces, although normally not considered a major concern with continuous fibre-reinforced ceramic composites, can introduce fabrication flaws which can have pronounced effects on the mechanical properties and behaviour (e.g. shape and level of the resulting load-displacement curve and shear strength). Machining damage introduced during test piece preparation can be either a random interfering factor in the determination of shear strength of pristine material or an inherent part of the strength characteristics to be measured. Universal or standardized test methods of surface preparation do not exist. Final machining steps can negate machining damage introduced during the initial machining. Thus, the history of the test piece fabrication can play an important role in the measured strength distributions and shall be reported.

5.4 Failures outside gauge section

Fractures that initiate outside the gauge section of a test piece can be due to extraneous stresses introduced by improper loading configurations or strength-limiting features in the microstructure of the test piece. Such non-gauge section fractures constitute invalid tests.

5.5 Thin test pieces

Thin test pieces (width to thickness ratio of more than 10) can suffer from splitting and instabilities, rendering, in turn, invalid test results.

6 Apparatus

6.1 Test machine

The test machine shall be equipped with a system for measuring the force applied to the test piece conforming to grade 1 or better according to ISO 7500-1.

6.2 Loading devices

The main purpose of the loading devices is to allow for uniform axial compression. Loading devices shall consist of rigid lower and upper rams with flat smooth faces vertical to axial force line.

6.3 Test fixture

The test fixture consists of upper and lower fixtures, loading pins and a loading ball, as shown schematically in [Figure 1](#). [Annex A](#) gives detailed information on the test fixture. The bottom surface of the lower fixture should be smooth and flat. The test piece is positioned between the upper and lower fixtures. The loading pins are placed at the contact points between each fixture and the test piece. The force is transmitted from the test machine to the fixture by the loading ball. If the test piece collapses at the contact positions with the loading pins, buffering spacers given in [A.3](#) can be inserted between the test piece and the loading pins. [Table 1](#) contains symbols, nomenclature and recommended dimensions for the test fixture ([Figure 1](#)), where the tolerances for L_a and L_i after assembly are $\pm 0,2$ mm. Centrality is the length equality between the centre of the fixture and each pin. [A.1](#) gives an example of the test fixture to satisfy the recommended dimensions in [Table 1](#). The tolerances for the diameters of the

loading ball and loading pins are $\pm 0,1$ mm and $\pm 0,01$ mm, respectively. The material of the test fixtures shall be uniform, isotropic, thermally stable and compatible with the test piece material at the test temperature.

NOTE 1 Silicon carbide is one of the representative materials to meet the requirements described in 6.3.

NOTE 2 When the buffering spacers are used, Figure 3 is not absolutely valid.

Table 1 — Recommended dimensions for test fixture

Dimension	Description	Value	Tolerance
L_i	Inner span	14,0 mm	$\pm 0,2$ mm
L_a	Outer span	56,0 mm	$\pm 0,2$ mm
	Centrality		$\pm 0,2$ mm
	Loading ball diameter	10,0 mm	$\pm 0,1$ mm
	Loading pin diameter	6,0 mm	$\pm 0,01$ mm

6.4 Heating apparatus

The heating apparatus shall provide a temperature control and measurement devices necessary to satisfy the requirement described in 9.3.2.

6.5 Temperature measurement

Temperature measurements of heating apparatus and a test piece shall be made with thermocouples that conform to IEC 60584-1 and IEC 60584-2. When connecting thermocouples to a test piece to measure its temperature directly, do not use thermocouples that cause damage or a chemical reaction.

6.6 Data recording system

A calibrated recorder can be used to record the force-displacement curve. The use of a digital data recording system combined with an analogue recorder is recommended.

Data recording system can record at least applied force and test machine displacement versus time. It also shall be accurate to ± 1 % of full scale and shall have a minimum data acquisition rate of 50 Hz.

6.7 Dimension-measuring devices

Micrometers and other devices used for measuring linear dimensions shall be accurate and precise to at least 0,01 mm and shall be in accordance with ISO 3611. To obtain consistent measurements of test piece dimensions, use a flat, anvil-type micrometer. Ball-tipped or sharp anvil micrometers are not recommended for woven continuous fibre-reinforced ceramic composites, because the resulting measurements can be affected by the peaks and valleys of the weave.

7 Test pieces

7.1 Test piece geometry

The required shape and tolerances of the asymmetric four-point bending test piece are shown in Figure 2. Table 2 contains recommended values for the dimensions of the test piece. These values depend on the test fixture to be used.

Table 2 — Recommended dimensions for asymmetric four-point bending test pieces

Dimension	Description	Value	Tolerance
l	Test piece length	76,0 mm	$\pm 0,1$ mm
L	Distance between notches	11,0 mm	$\pm 0,1$ mm
b	Test piece width	19,0 mm	$\pm 0,1$ mm
R	Notch radius	1,3 mm	—
θ	Notch angle	90,0°	—
H	Test piece thickness	$\geq 3,0$ mm	—

7.2 Test piece preparations

During cutting out, care shall be taken to prevent damage to the materials and to align the test piece axis with the desired fibre orientation. The test piece axis with the fibre orientation shall be recorded.

Notches of a test piece should be machined with grinding or cutting method as carefully as possible. In machining notches, care shall be taken to prevent burr, trim or chipping on edges.

7.3 Number of specimen tests

A minimum of five valid test results is recommended for the purpose of estimating a mean.

8 Test conditions

8.1 Test modes and rates

8.1.1 General

Test modes may involve load or displacement control. Recommended rates of testing shall be sufficiently rapid to obtain the maximum possible shear strength at fracture of the material within 30 s. However, rates other than those recommended here may be used to evaluate rate effects. In all cases, the test mode and rate shall be reported.

Generally, displacement-controlled tests are employed in such cumulative damage or yielding deformation processes to prevent a 'runaway' condition (i.e. rapid uncontrolled deformation and fracture) characteristic of load- or stress-controlled tests. However, for sufficiently rapid test rates, differences in the fracture process cannot be noticeable and any of these test modes can be appropriate.

8.1.2 Displacement rate

Use a constant cross-head displacement rate of 0,05 mm/s, unless otherwise found acceptable as determined in [8.1.2](#).

8.1.3 Load rate

Select a constant loading rate to produce final fracture in 5 s to 30 s or to be approximately equivalent to a test rate of 0,05 mm/s.

8.2 Test temperature

The test temperature shall be measured in at least two locations in accordance with [6.5](#). One is the centre of the test piece on the front side and the other is the centre of the test piece on the back side. The test temperature shall meet the requirement in [9.3.2](#).

9 Procedures

9.1 Test piece dimensions

Determine the thickness and distance between notches in the gauge section of each test piece to within 0,01 mm. Avoid damaging the critical gauge-section area by performing these measurements either optically (e.g. using an optical comparator) or mechanically using a flat, anvil-type micrometer. In either case, the resolution of the instrument shall be as specified in 6.7. Exercise extreme caution to prevent damaging the gauge section of the test piece. Record and report the measured dimensions and locations of the measurements for use in the calculation of the shear stress. Use the average of multiple measurements in the stress calculations.

9.2 Test preparation

Set the test mode and test rate on the test machine. Set the autograph data acquisition systems ready for data logging.

Zero the load cell.

9.3 Test implementation

9.3.1 Mounting of test piece on test fixture

Mount the lower fixture on the centre of the lower ram. Mount the loading pins on the lower fixture. Position the test piece on the loading pins. Place the upper fixture with two loading pins on the test piece. Align the notch of the test piece to the centre of both upper and lower fixtures. Alignment tool 2, shown in A.2, is effective to centre the test piece V-notch relative to the fixture horizontally. The schematic of the fixture after assembly is shown in Figure 1. Place the loading ball on the centre of the top surface of the upper fixture. Bring the upper ram close to the loading ball to prepare for testing. Apply a small amount of pre-loading (20 N to 50 N).

If it is difficult to assemble the fixtures, the upper fixture and the loading pins should be tied with rubber bands or strings temporarily. Untie them after pre-loading.

To obtain the accurate shear strength, the test piece shall be set normal to the upper and lower fixtures. A back plate, shown in A.2, is effective as alignment tool 1.

9.3.2 Heating of test piece

Begin to record the test temperature. Heat the test piece to the required test temperature and maintain this temperature for a period to allow for temperature stabilization.

The hold time depends on the time necessary to ensure that the test piece has reached thermal equilibrium. Maintain the test piece temperature within the limits specified in Table 3 during the time for temperature stabilization. Report the hold time.

Table 3 — Allowable test temperature variation

Test temperature, T	Variation
< 1 000°C	±3°C
≥ 1 000°C	±6°C

There are two ways to regulate heating, as follows:

- If test piece temperature is measured during the test on the test piece itself, this temperature should be used to control the furnace.

- If it is not possible to measure the test piece temperature directly during the test, then it is necessary to use the relationship between the test piece temperature and the control temperature of the furnace. In this case, calibration is necessary. The relationship between the test piece temperature at the centre of the gauge section and the control temperature shall be established beforehand on a dummy test piece over the range of temperature of interest.

Ensure that the test piece stays in the initial state of stress during heating through a load-controlled mode with pre-load value (between 20 N and 50 N, as explained in [9.3.1](#)).

The difference between the indicated temperature and the nominal test temperature shall not exceed the values specified in [Table 3](#).

Care shall be taken to prevent damage to a test piece.

Care shall be taken to ensure that the temperature overshoot during heating does not exceed the range specified in [Table 3](#).

To prevent a test piece from overloading due to thermal expansions of the test piece and apparatus, heat the test piece under the load-controlled mode. The amount of pre-load should not exceed 5 % of the expected failure load.

9.3.3 Loadings

Initiate the data collection. Apply the load to the test piece at the specified rate until failure.

9.4 Completion of testing

After test piece fracture, disable the action of the test machine and the data collection of the data acquisition system. The breaking load should be measured with an accuracy of $\pm 1\%$ of the load range and noted for the report. Carefully remove the test piece halves from the fixtures. Avoid damaging the fracture surfaces by preventing them from touching each other or other objects. [Figure 4](#) shows the gauge section of asymmetric four-point bending specimen after a test.



Figure 4 — Gauge section of specimen after testing

9.5 Test validity

The following circumstances invalidate a test:

- failure to specify and record test conditions;
- failure outside the gauge section of the test piece.

NOTE If a test piece collapses at the loading pins, a spacer as described in [A.3](#) is effective in preventing damage.

10 Calculation of results

10.1 Shear strength

Calculate the shear strength, τ_m , according to [Formula \(1\)](#).

$$\tau_m = \frac{F_{\max} (L_a - L_i)}{S_0 (L_a + L_i)} \quad (1)$$

where

F_{\max} is the shear failure force (N);

L_a and L_i are the outer and inner spans (mm), respectively;

S_0 is the initial gauge section (mm²), calculated according to [Formula \(2\)](#).

$$S_0 = H \cdot L \quad (2)$$

where

H is the test piece thickness (mm);

L is the distance between the notches (mm).

NOTE The shear force distribution caused by the applied force F is shown in [Figure 3 C](#).

10.2 Error calculation

For each series of tests, calculate the mean, \bar{X} , standard deviation, s , and coefficient of variation, C_V , for the shear strength according to [Formulae \(3\)](#), [\(4\)](#) and [\(5\)](#), respectively.

$$\bar{X} = \frac{1}{n} \sum_{i=1}^n X_i \quad (3)$$

$$s = \sqrt{\frac{1}{n-1} \sum_{i=1}^n (X_i - \bar{X})^2} \quad (4)$$

$$C_V = \frac{100(s)}{\bar{X}} \quad (5)$$

where

X_i represents the i -th measured value;

n is the number of valid tests.

11 Test report

The test report shall contain the following information:

- a) a reference to this document, i.e. determined in accordance with ISO 19587:2021;
- b) the name and address of the testing establishment;
- c) the date of the test, unique identification of report and of each page, customer name and address and signatory;
- d) a test piece drawing or reference;
- e) a description of the test material (material type, manufacturing code, batch number, reinforcement directions of the material with respect to the longitudinal axis of the test piece);
- f) a description of the test set-up: heating system, test frame type, load cell and temperature measurement device;
- g) test environment, including temperature and atmosphere;
- h) pre-load level, test mode (load or displacement control), actual test rate (load or displacement rate), heating rate, hold time and test temperatures at the beginning of the test;
- i) the number of tests carried out and the number of valid results obtained;
- j) a force-test machine displacement diagram;
- k) the mean, standard deviation and coefficient of variation for the measured shear strength for each test series;
- l) the failure location and appearance of the test piece after fracture;
- m) if a buffering spacer is used, its material and geometry;
- n) any significant deviations from the procedures and requirements of this test method.

Annex A (informative)

A.1 Fixture drawing

An example of the fixture that specifies the condition in [Table 1](#) is shown in [Figure A.1](#).

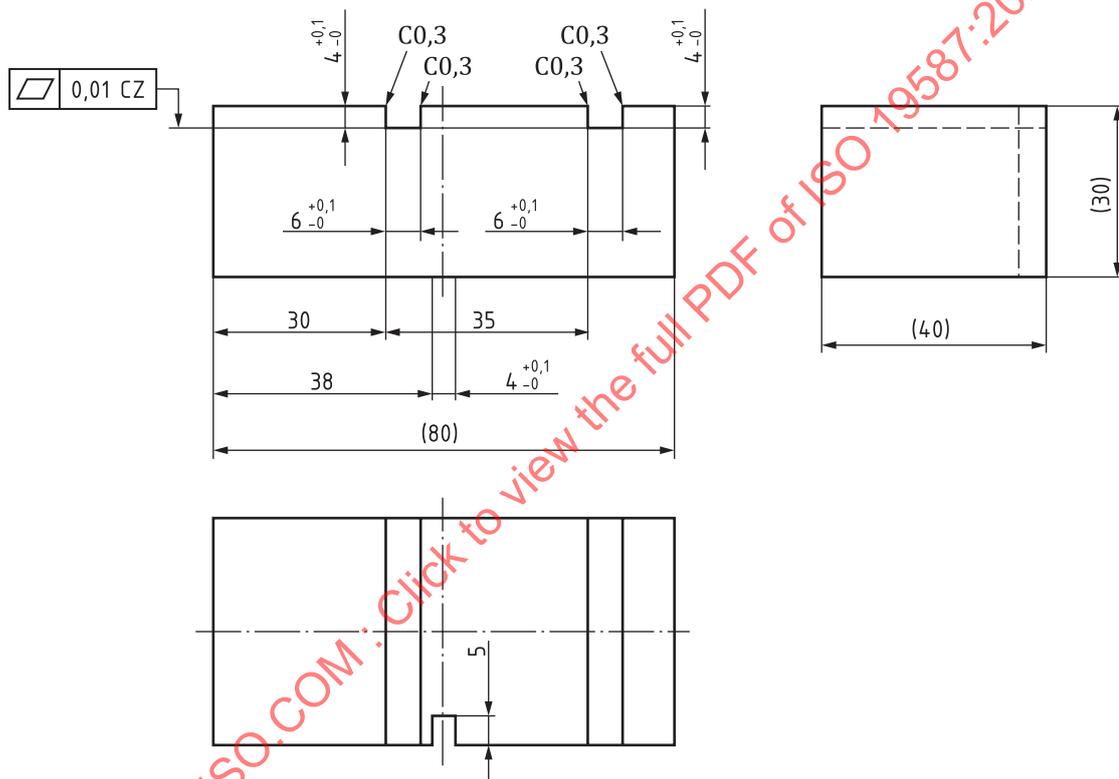


Figure A.1 — Example of fixture drawing

A.2 Alignment tool

Alignment tools as shown in [Figure A.2](#) are effective to align the test piece and fixtures.

Alignment tool 1 is a tool to align the upper and lower fixtures vertically and place the test piece in the width centre. Place alignment tool 1 on the lower loading pins. Mount the test piece and place the upper fixture with the loading pins on the test piece. Press the test piece and the upper and lower fixtures against alignment tool 1 as shown in [Figure A.3](#) so as to align them vertically.

Alignment tool 2 is a tool to centre the test piece V-notch relative to the fixtures horizontally. After assembling the fixtures, insert alignment tool 2 in the grooves of the lower and upper fixtures. Pull alignment tool 2 vertically up into the V-notch.