
**Fine ceramics (advanced ceramics,
advanced technical ceramics) —
Determination of the interlaminar shear
strength of continuous-fibre-reinforced
composites at ambient temperature by
the compression of double-notched test
pieces and by the losipescu test**

*Céramiques techniques — Détermination de la résistance au
cisaillement interlaminaire des composites renforcés de fibres connues
à température ambiante par compression d'éprouvettes doublement
entaillées et par l'essai de losipescu*



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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 20505 was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of the interlaminar shear strength of continuous-fibre-reinforced composites at ambient temperature by the compression of double-notched test pieces and by the Iosipescu test

1 Scope

This International Standard specifies a method for the determination of interlaminar shear strength of continuous-fibre-reinforced ceramic composites at ambient temperature, by the compression of a double-notched test piece or by the Iosipescu test. Methods for test piece fabrication, testing modes and rates (load rate or displacement rate), data collection, and reporting procedures are addressed.

This International Standard applies primarily to advanced ceramic or glass-matrix composites with continuous-fibre reinforcement having uni-directional (1-D) or bi-directional (2-D) fibre architecture. This test method does not address composites with (3-D) fibre architecture or discontinuous-fibre-reinforced, whisker-reinforced or particulate-reinforced ceramics.

NOTE 1 Values expressed in this International Standard are in accordance with the International System of Units (SI).

NOTE 2 This International Standard is based on ASTM C1292.

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, *Micrometer callipers for external measurement*

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

ASTM C1292, *Standard Test Method for Shear Strength of Continuous Fiber-Reinforced Advanced Ceramics at Ambient Temperatures*

3 Terms and definitions

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

3.1

fine ceramic (advanced ceramic, advanced technical ceramic)

highly engineered, high-performance predominately non-metallic, inorganic, ceramic material having specific functional attributes

3.2
continuous-fibre-reinforced ceramic composite
CFCC
 ceramic matrix composite in which the reinforcing phase consists of a continuous fibre, continuous yarn, or a woven fabric

3.3
shear-failure load
 maximum load required to fracture a shear-loaded test piece

3.4
shear strength
 maximum shear stress which a material is capable of sustaining

NOTE Shear strength is calculated from the shear-fracture load and the shear-loaded area.

4 Symbols and abbreviated terms

The symbols used throughout this International Standard and their designations are given in Table 1.

Table 1 — Symbols and designations

Symbol	Designation	Unit	References
L	Test piece length	mm	Tables 2, 3
h	Distance between notches	mm	Tables 2, 3 Equations 2, 4
w	Test piece width	mm	Tables 2, 3 Equation 2
t	Test piece thickness	mm	Tables 2, 3 Equation 4
d	Notch width, double-notched test piece	mm	Table 2
R	Notch radius, Iosipescu test piece	mm	Table 3
θ	Notch angle, Iosipescu test piece	°	Table 3
n	Number of valid tests	1	Equations 5, 6 subclause 12.2
$P_{\max}$	Maximum load	N	Equations 1, 3
A_1	Shear area for double-notched test piece	mm ²	Equations 1, 2
A_2	Shear area for Iosipescu test piece	mm ²	Equations 3, 4
τ_{IL}	Interlaminar shear strength	MPa	Equations 1, 3
$\bar{X}$	Mean value	MPa	Equations 5, 6, 7
SD	Standard deviation	MPa	Equations 6, 7
CV	Coefficient of variation	1	Equation 8

5 Principle

This International Standard is for material development, material comparison, quality assurance, characterization, reliability and design data generation. The interlaminar shear strength of continuous-fibre-reinforced ceramic composites, as determined by this International Standard, can be measured by the compression of double-notched test pieces or by the Iosipescu test. In the case of the former, a double-notched test piece of uniform width is loaded in compression to induce failure by shear between two centrally located notches machined halfway through the thickness and spaced a fixed distance apart on opposing faces. Schematics of the test setup and the test piece are shown in Figures 1 and 2.

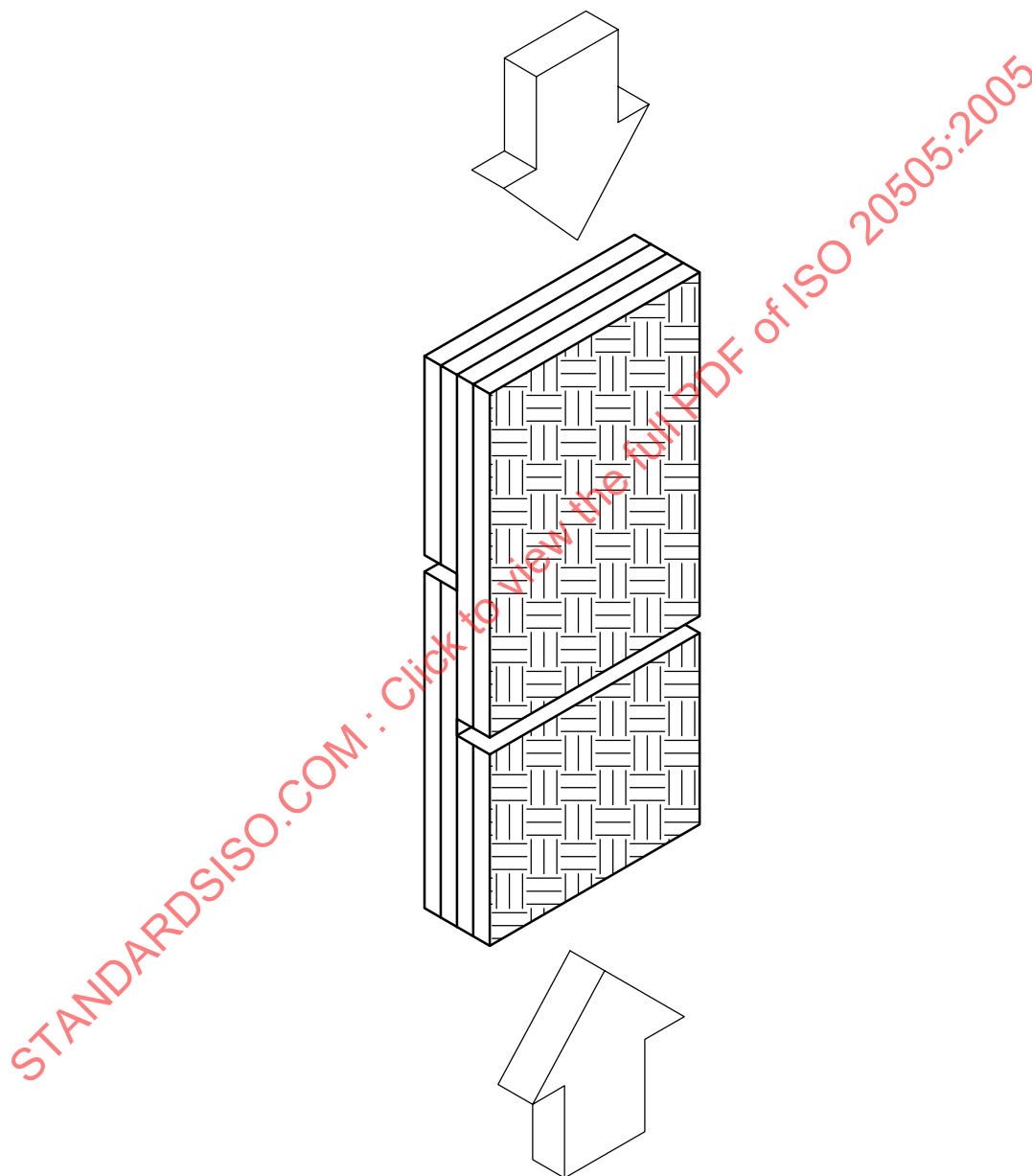


Figure 1 — Schematic of double-notched test piece subjected to compressive loading

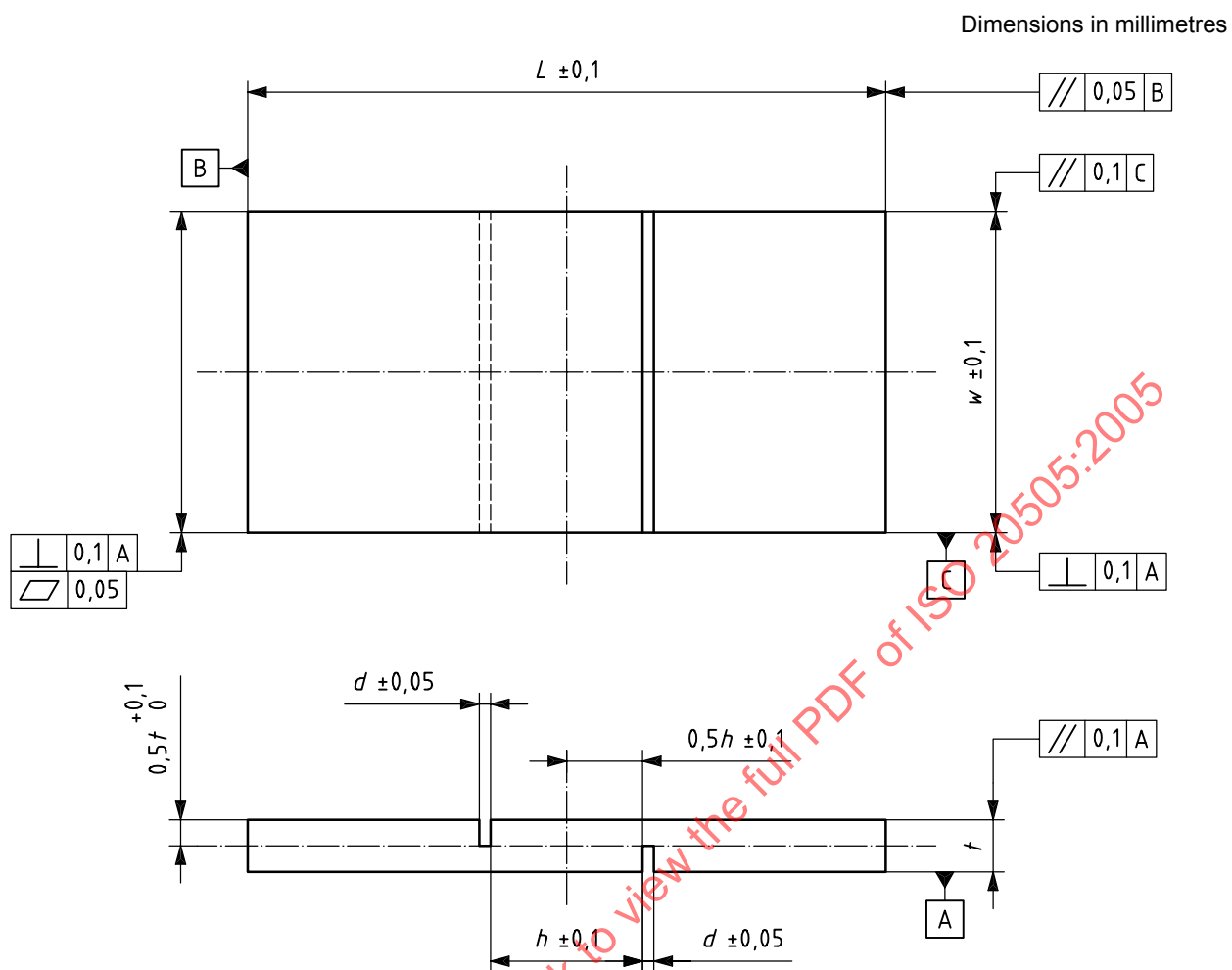


Figure 2 — Geometry and dimensions of double-notched test piece

For the Iosipescu 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 between the V-notches. Schematics of the test setup and test piece are shown in Figures 3 and 4.

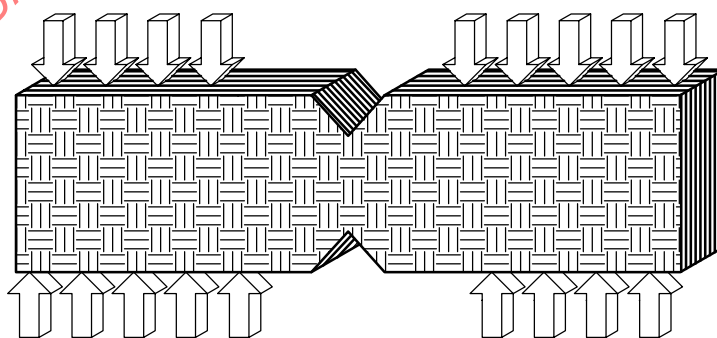


Figure 3 — Schematic of Iosipescu test piece subjected to asymmetric four-point bending loading

Dimensions in millimetres

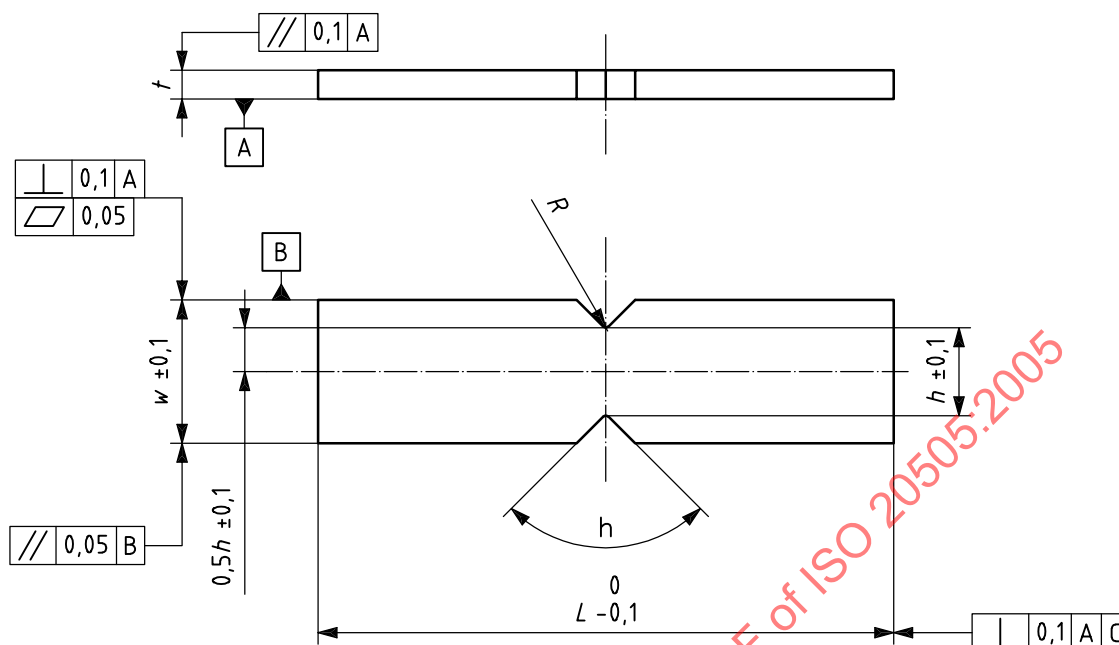


Figure 4 — Geometry and dimensions of losipescu test piece

6 Interferences

6.1 Test environment

The test environment may 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 and/or at sufficiently rapid testing rates, 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, relative humidity and temperature shall be monitored and reported.

6.2 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 may 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 may, or may not, negate machining damage introduced during the initial machining. Thus, the history of test piece fabrication may play an important role in the measured strength distributions and shall be reported.

6.3 Bending

Bending of uniaxially loaded shear test pieces (during the compression of double-notched test pieces) can cause or promote non-uniform stress distributions that may alter the desired uniform state of stress during the test.

6.4 Failures outside gauge section

Fractures that initiate outside the uniformly stressed gauge section of a test piece may 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 will constitute invalid tests.

6.5 Notch separation

For the evaluation of the interlaminar shear strength by the compression of a double-notched test piece, the distance between the notches has an effect on the maximum load and therefore on the interlaminar shear strength. It has been found that the stress distribution in the test piece is independent of the distance between the notches when the notches are far apart. However, when the distance between the notches is such that the stress fields around the notches interact, the measured interlaminar shear strength increases. Because of the complexity of the stress field around each notch and its dependence on the properties and homogeneity of the material, it is recommended to perform a series of tests on test pieces with different spacing between the notches, to determine their effect on the measured interlaminar shear strength.

6.6 Specimen clamping

Because the purpose of the jaws is to maintain the test piece in place and to prevent buckling, excessive clamping force with the jaws of the fixture during the compression of double-notched test pieces will reduce the stress concentration around the notches and therefore artificially increase the measured interlaminar shear strength. In the case of the Iosipescu fixture, avoid over-tightening the jaws because it induces undesirable pre-loading and may damage some materials.

6.7 Friction

Many fixtures for both the compression of double-notched test pieces and the Iosipescu test incorporate an alignment mechanism in the form of a guide rod and a linear roller bearing. Excessive free play or excessive friction in this mechanism may introduce spurious moments that will alter the ideal loading conditions.

7 Apparatus

7.1 Testing machines

The testing machine shall be verified in accordance with ISO 7500-1 and shall be at least grade 1,0.

7.2 Data acquisition

Obtain at least an autographic record of applied load and cross-head displacement versus time, using either analogue chart recorders or digital data acquisition systems. Recording devices shall be accurate to within $\pm 1\%$ of the selected range for the testing equipment including readout unit, and have a minimum data acquisition rate of 10 Hz with a response of 50 Hz deemed more than sufficient.

7.3 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 may be affected by the peaks and valleys of the weave. Measure test piece dimensions to within 0,02 mm.

7.4 Test fixtures

There are various types of fixtures for the compression of double-notched test pieces. One type consists of a stationary element mounted on a base plate, an element that attaches to the cross-head of the testing machine, and two jaws to fix the test piece in position. A schematic description of such a test fixture is shown in Figure 5. Another type is a simple anti-buckling test fixture, where the test piece is held in position using a plate that clamps the test piece against a stationary element mounted on a base plate. Figure 6 shows a schematic of such a fixture. For the losipescu test, a modified asymmetric four-point bending fixture is recommended. This fixture consists of a stationary element mounted on a base plate, and a movable element capable of vertical translation guided by a stiff post. The movable element is attached to the cross-head of the testing machine. Each element clamps half of the test piece into position, with a wedge-action grip that is able to compensate for minor variations in test piece width. A span of 13 mm is left unsupported between fixture halves. An alignment tool is recommended to ensure that the test piece notch is aligned with the line-of-action of the loading fixture. Figure 7 shows a photograph of a commercially available losipescu fixture, while Figure 8 shows a schematic of it.

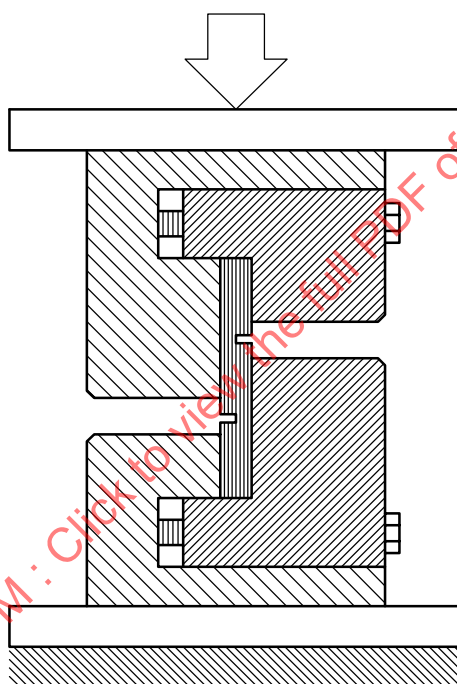


Figure 5 — Example of anti-buckling fixture for compression of double-notched test piece

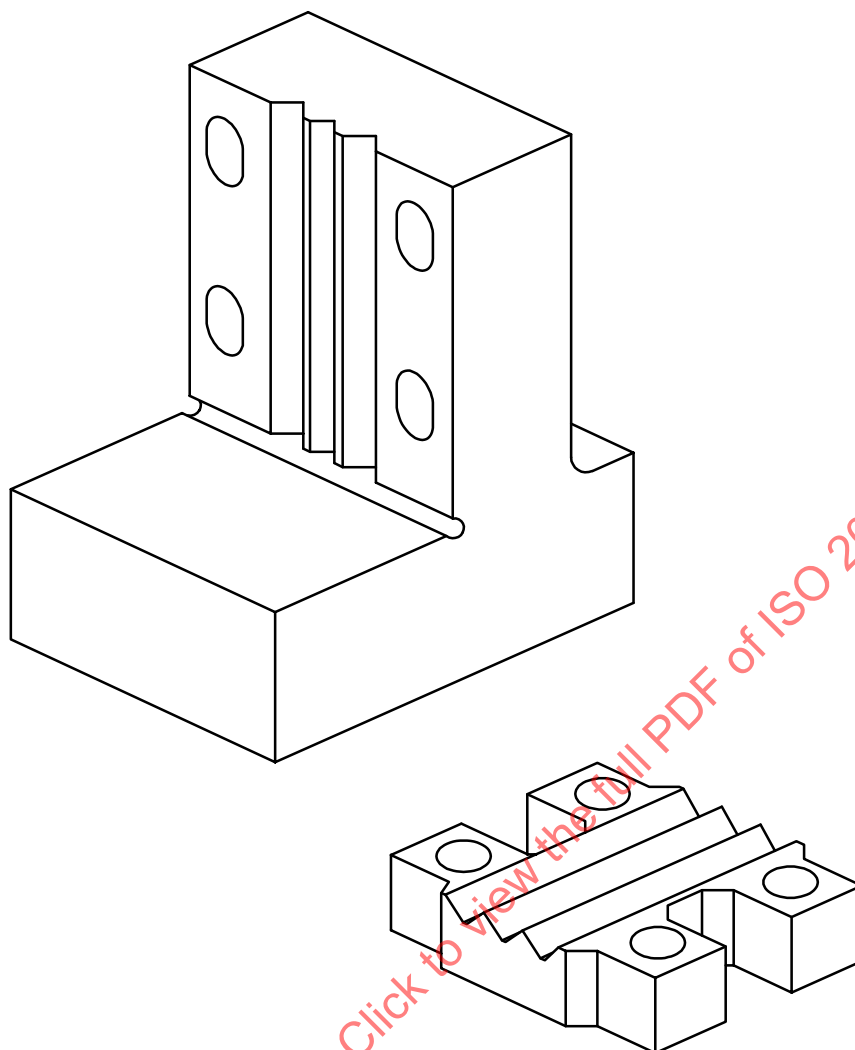
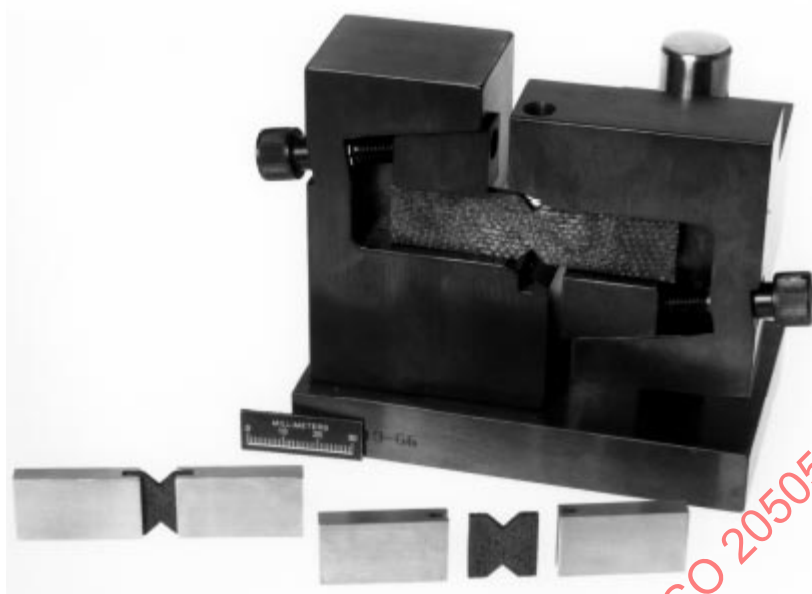
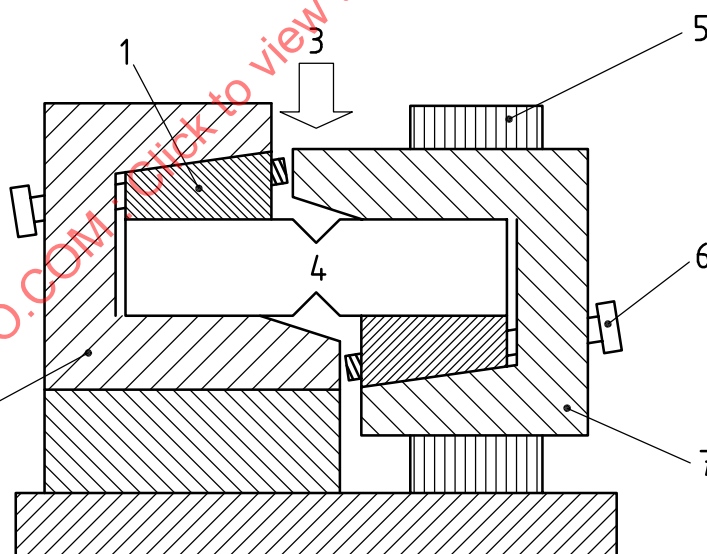


Figure 6 — Example of anti-buckling fixture for compression of double-notched test piece



The test pieces in the foreground illustrate the use of adhesively bonded end-tabs for evaluating specimens obtained from thin plates.

Figure 7 — Photograph of commercially available fixture for losipescu test



Key

- | | |
|--|---|
| 1 adjustable wedge to tighten the specimen | 5 fixture guide rod |
| 2 stationary portion of fixture | 6 wedge-adjusting screw |
| 3 load | 7 fixture attached to guide rod by linear rolling bearing |
| 4 specimen | |

Figure 8 — Schematic of losipescu test fixture

8 Test piece

8.1 Test piece geometry

8.1.1 Double-notched test piece

Double-notched test pieces shall conform to the shape and tolerances shown in Figure 2. The double-notched test piece consists of a rectangular plate with notches machined on both sides. The depth of the notches shall be at least equal to one-half of the test piece thickness, and the distance between the notches shall be determined considering the requirements to produce shear failure in the gauge section. Furthermore, because the measured interlaminar shear strength may be dependent on the notch separation, it is recommended to perform tests with different values of notch separation to determine this dependence. The edges of the test pieces shall be smooth, but not rounded or bevelled. Table 2 contains recommended values for the dimensions associated with the test piece shown in Figure 2.

Table 2 — Recommended dimensions for double-notched compression test pieces

Values in millimetres

Dimension	Description	Value	Allowance
L	Test piece length	30,00	$\pm 0,1$
h	Distance between notches	6,00	$\pm 0,1$
w	Test piece width	15,00	$\pm 0,1$
d	Notch width	0,50	$\pm 0,05$
	Notch depth	$t/2$	$+ 0,01/- 0,0$
t	Test piece thickness	—	

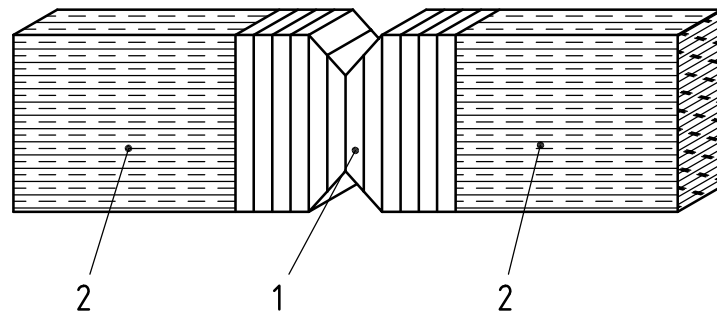
8.1.2 Iosipescu test piece

The required shape and tolerances of the Iosipescu test piece are shown in Figure 4, while Table 3 contains recommended values for the dimensions of the test piece. If required, the dimensions of the test piece, particularly the notch angle, notch depth and notch radius, may be adjusted to meet special material requirements, but any deviation from the recommended values listed in Table 3 shall be reported with the test results, although the standard tolerances shown in Figure 4 still apply.

Because, in some instances due to limitations in material processing, it may be difficult to produce thick sections to conform with the dimensions and geometry shown in Table 3 and Figure 4, respectively, the test piece geometry may be modified to obtain appropriate results. In this case, adhesively bonded end-tabs may be used, and the depth and angle of the notches shall be selected to promote shear failure between the V-notches. Figure 9 shows an example of this situation (such a specimen is shown in the foreground in Figure 7).

Table 3 — Recommended dimensions for Iosipescu test pieces

Dimension	Description	Value	Allowance
L	Test piece length	76,00 mm	$\pm 0,1$
h	Distance between notches	11,00 mm	$\pm 0,1$
w	Test piece width	19,00 mm	$\pm 0,1$
R	Notch radius	0,50 mm	
θ	Notch angle	90°	
t	Test piece thickness	—	

**Key**

- 1 specimen
- 2 adhesively bonded end-tab

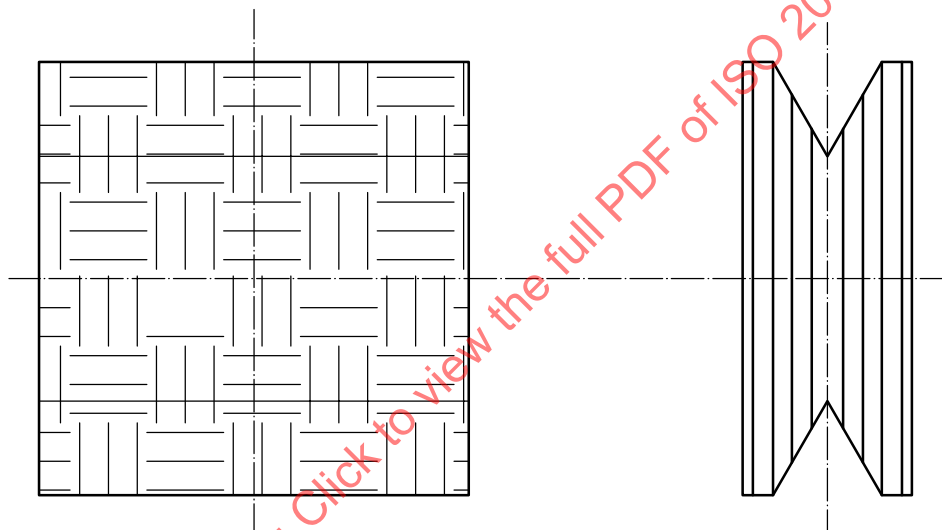


Figure 9 — Schematic of short Iosipescu test piece

8.2 Test piece preparation

8.2.1 Customary practices

In instances where a customary machining procedure has been developed that is completely satisfactory for a class of materials (that is, it induces no unwanted surface/subsurface damage or residual stresses), this procedure shall be used.

8.2.2 Standard procedures

Studies to evaluate the machinability of continuous-fibre reinforced ceramic composites have not been completed. Therefore, the standard procedures of this subclause can be viewed as starting-point guidelines, but a more stringent procedure may be necessary.

All grinding or cutting shall be done with an ample supply of appropriate filtered coolant, to keep the workplace and grinding wheel constantly flooded and particles flushed. Grinding can be done in at least two stages, ranging from coarse to fine rate of material removal.

The stock removal rate shall be on the order of 0,03 mm per pass, using diamond tools that have between 320 and 600 grit. Remove equal amounts of stock from each face where applicable.

8.2.3 Handling precautions

Exercise care in the storing and handling of finished test pieces to avoid the introduction of severe flaws. In addition, direct attention to pre-test storage of test pieces in controlled environments or desiccators, to avoid unquantifiable environmental degradation of test pieces prior to testing.

8.3 Number of test pieces

A minimum of 5 valid test results is required for the purpose of estimating a mean value. A greater number of tests may be necessary, if estimates regarding the form of the strength distribution are required.

9 Precautionary statement

During the conduct of this test method, the possibility of flying fragments of broken test material may be high. The brittle nature of advanced ceramics and the release of strain energy contribute to the potential release of uncontrolled fragments upon fracture. Means for containment and retention of these fragments for later fractographic reconstruction and analysis is highly recommended.

WARNING — Exposed fibres at the edges of continuous-fibre-reinforced ceramic composite test pieces present a hazard due to the sharpness and brittleness of the ceramic fibres. All persons required to handle these materials must be well informed of these conditions and the proper handling techniques.

10 Test conditions

10.1 Test modes and rates

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 may not be noticeable and any of these test modes may be appropriate.

10.1.1 Displacement rate

Use a constant cross-head displacement rate of 0,05 mm/s, unless otherwise found acceptable as determined under the conditions specified in 10.1.2.

10.1.2 Load rate

Select a constant loading rate to produce final fracture in 10 s to 30 s, or to be approximately equivalent to a test rate of 0,05 mm/s.

11 Procedure

11.1 Test piece dimensions

For double-notched test pieces, determine the thickness and width of the gauge section of each test piece to within 0,02 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 7.3. 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.

Additionally, post-fracture measurements of the gauge section dimensions shall be made using the instruments described above. In the case of post-fracture measurements, only the dimensions at the plane of fracture shall be measured and recorded for the purpose of calculating the shear strength. Note that, in some cases, the fracture process can severely fragment the gauge section thus making post-fracture measurements of dimensions difficult. In these cases, the procedures outlined in 10.1 shall suffice.

11.2 Preparations for testing

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

11.3 Conducting the test

11.3.1 Mount the test piece in the test fixture.

11.3.1.1 Compression of double-notched test pieces

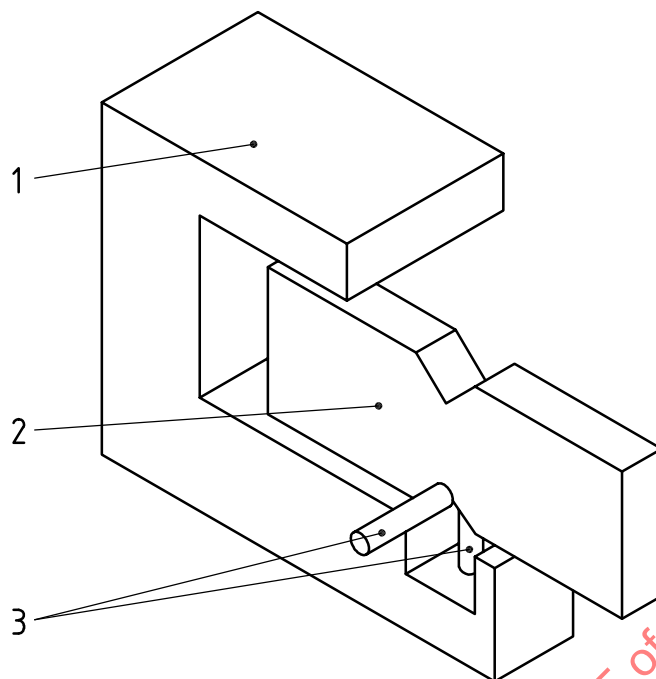
Loosen the jaw of each grip sufficiently to allow the test piece thickness to be freely inserted into the fixture with clearance. Place the test piece loosely in the centre of the fixture and then press the back side of the test piece against the back wall of the fixture, while aligning the bottom of the test piece against the bottom of the fixture. The test piece should be centered in the fixture so that the line-of-action of the load acts directly through the mid-plane of the test piece. Lightly tighten the jaws to fix the test piece in the fixture. DO NOT OVER-TIGHTEN THE JAWS. The purposes of the jaws are to maintain the test piece in place and to prevent buckling, not for clamping. Over-tightening the jaws will result in artificially high shear strengths. Slowly move the cross-head of the testing machine until the upper surface of the fixture just contacts the upper surface of the test piece.

11.3.1.2 Iosipescu test pieces

Loosen the jaw of each grip sufficiently to allow the specimen width to be freely inserted into the grip with clearance. Adjust the movable head position until the grips are approximately aligned vertically. Place the specimen loosely into both grips. Press the back side of the specimen flat against the back wall of the fixture. Pull the specimen alignment tool vertically up into the notch to centre the specimen V-notch relative to the fixture in accordance with Figure 10. While keeping the specimen centered, lightly tighten the left-hand-side jaw on the lower grip. DO NOT OVER-TIGHTEN THE JAW; over-tightening induces undesirable pre-loading and may damage some materials. There should now be some clearance between the specimen and the upper grip and no load showing in the test machine. If there is no clearance, or if load in the specimen is indicated, adjust either the head or the jaw of the upper grip, or both, until there is both clearance and zero load. Re-check the specimen placement in the lower grip. Repeat if necessary. Move the testing machine cross-head until the upper surface of the upper grip just contacts the upper surface of the right-hand side of the specimen, without loading it. Lightly tighten the jaw of the upper, right-hand, grip onto the right-hand side of the specimen. DO NOT OVER-TIGHTEN THE JAW; over-tightening induces undesirable pre-loading and may damage some materials. Pre-loading should be minimized, however a small amount of pre-loading (20 N to 50 N) may be unavoidable. The specimen should now be centered in the fixture so that the line-of-action of the load acts directly through the centre of the notch on the specimen.

11.3.2 Begin data acquisition.

Initiate the action of the test machine.



Key

- 1 fixture
- 2 specimen
- 3 alignment tool

Figure 10 — Schematic of alignment tool for Iosipescu test.

11.4 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 test piece mount and determine the dimensions of the failed sheared area to the nearest 0,02 mm, by measurement of this surface with respect to either half of the ruptured test piece. This technique affords the most accurate determination of the length of the sheared plane defined by the separation of the notches machined in the test piece. Avoid damaging the fracture surfaces by preventing them from contacting each other or other objects. Figure 11 shows a micrograph of the edge of a double-notched test piece after the test.

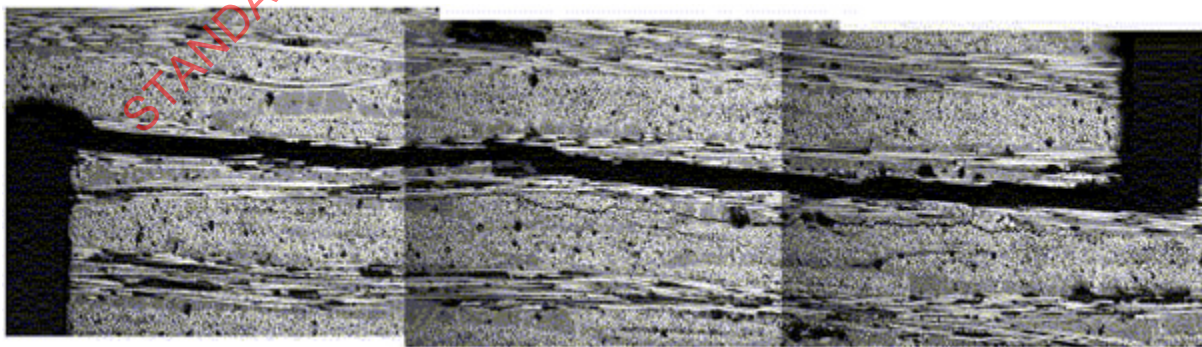


Figure 11 — Micrograph of double-notched test piece after the test

11.5 Post test

Determine the ambient temperature and relative humidity. Measure and report the fracture location. Note that the use of results from test pieces fracturing outside the uniformly stressed gauge section cannot be used in the direct calculation of mean shear strength. Results from test pieces fracturing outside the gauge section are considered anomalous and can be used only as censored tests. To complete a required statistical sample for purposes of average strength, one replacement test piece should be tested for each test piece which fractures outside the gauge section. Visual examination and light microscopy are recommended to determine the mode and type of fracture, as well as the location of fracture initiation.

12 Calculation of results

12.1 Shear strength

12.1.1 Double-notched test piece

For the compression of double-notched test pieces, calculate the shear strength as:

$$\tau_{IL} = \frac{P_{\max}}{A_1} \quad (1)$$

where

$P_{\max}$ is the applied maximum load;

A_1 is the shear stressed area, which is calculated as:

$$A_1 = wh \quad (2)$$

where

w is the width of the test piece;

h is the distance between the notches (Figure 2).

12.1.2 Iosipescu test piece

For Iosipescu test pieces, calculate the shear strength as:

$$\tau_{IL} = \frac{P_{\max}}{A_2} \quad (3)$$

where

$P_{\max}$ is the applied maximum load;

A_2 is the shear stressed area, which is calculated as:

$$A_2 = th \quad (4)$$

where

t is the thickness of the test piece;

h is the distance between the notches (Figure 4).

12.2 Statistics

For each series of tests, calculate the mean, standard deviation and coefficient of variation for the interlaminar shear strength as follows:

$$\text{Mean} \quad \bar{X} = \frac{1}{n} \sum_{i=1}^n X_i \quad (5)$$

$$\text{Standard deviation SD} \quad \text{SD} = \sqrt{\frac{\sum_{i=1}^n (X_i - \bar{X})^2}{n-1}} \quad (6)$$

$$\text{Coefficient of variation} \quad \text{CV} = \frac{100(\text{SD})}{\bar{X}} \quad (7)$$

where

X_i represents the i -th measured value;

n is the number of valid tests.

13 Test report

The test report shall include the following information:

- a) date and location of testing;
- b) test piece geometry used (include engineering drawing);
- c) a drawing or sketch of the type and configuration of the test machine; if a commercial test machine is used, the manufacturer and model number of the test machine will suffice;
- d) a drawing or sketch of the type and configuration of the test piece mount;
- e) the total number of test pieces (n) with special emphasis on the number of test pieces that fractured in the gauge section; this information will reveal the success rate of the particular test piece geometry and test apparatus;
- f) all relevant data such as vintage and identification data, with emphasis on the date of manufacture of the material and a short description of reinforcement (type, lay-up, etc), fibre volume fraction and bulk density; for commercial materials, the commercial designation shall be reported;
- g) for non-commercial materials, the major constituents and proportions, as well as the primary processing route including green state and consolidation routes; also the fibre volume fraction, matrix porosity, and bulk density;
- h) description of the method of test piece preparation, including all stages of machining;
- i) heat treatments, coatings, or pre-test exposures, if any are applied either to the as-processed material or to the as-fabricated test piece;
- j) test environment including relative humidity, temperature, and atmosphere (e.g. ambient air, dry nitrogen, silicone oil, etc.);