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Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of the in-plane shear strength of continuous-fibre-reinforced composites at ambient temperature by the Iosipescu test

Céramiques techniques — Détermination de la résistance au cisaillement plan des composites renforcés de fibres continues à température ambiante par l'essai de Iosipescu

Reference number
ISO 20506:2005(E)

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Foreword

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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 20506 was prepared by Technical Committee ISO/TC 206, *Fine ceramics.*

INTERNATIONAL STANDARD **ISO 20506:2005(E)**

Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of the in-plane shear strength of continuous-fibre-reinforced composites at ambient temperature by the Iosipescu test

1 Scope

This International Standard specifies a method for the determination of in-plane shear strength of continuous-fibre-reinforced ceramic composites at ambient temperature 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), bi-directional (2-D) or 3-D fibre architecture. This test method does not address composites with 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 designations

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	Table 2
h	Distance between notches	mm	Table 2 Equation 2
w	Test piece width	mm	Table 2
t	Test piece thickness	mm	Table 2 Equation 2
R	Notch radius	mm	Table 2
θ	Notch angle	°	Table 2
n	Number of valid tests	1	Equations 3, 4
P_{max}	Maximum load	N	Equation 1
A	Shear area of test piece	mm^2	Equation 1
τ_{IP}	In-plane shear strength	MPa	Equation 1
$\overline{X}$	mean	MPa	Equation 3, 4, 5
SD	standard deviation	MPa	Equation 4
CV	Coefficient of variation	1	Equation 5

5 Principle

This International Standard is for material development, material comparison, quality assurance, characterization, reliability and design data generation. The in-plane shear strength of continuous-fibre-reinforced ceramic composites, as determined by this International Standard, is measured by the Iosipescu test. 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 a modified asymmetric four-point bending fixture. Failure of the test piece occurs by shear between the V-notches. Schematics of the test setup and the test piece are shown in Figures 1 and 2.

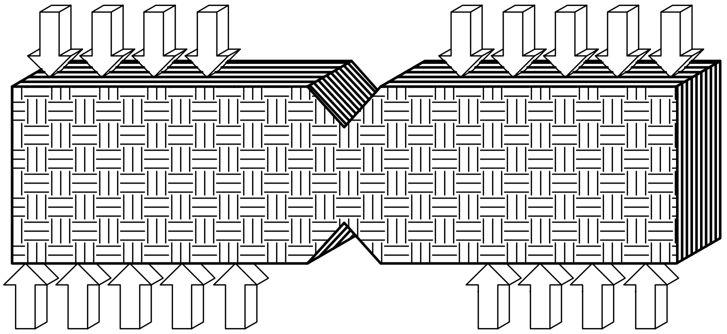


Figure 1 — Schematic of Iosipescu test piece subjected to asymmetric four-point bending

Dimensions in millimetres

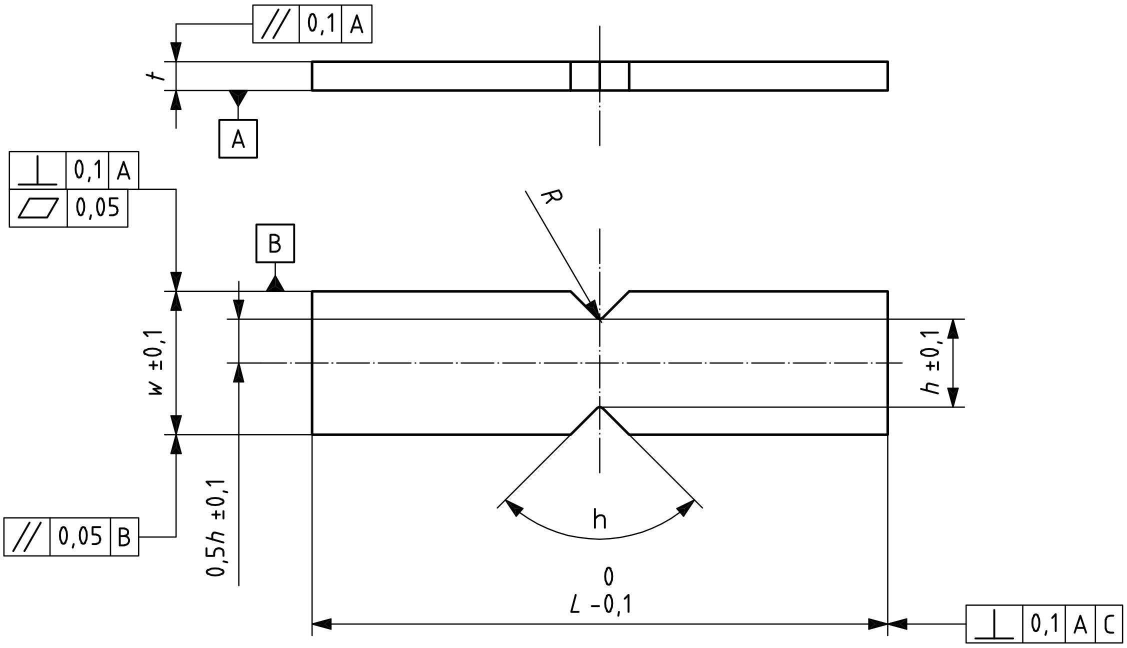


Figure 2 — Geometry and dimensions of Iosipescu test piece

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 the test piece fabrication may play an important role in the measured strength distributions and shall be reported.

6.3 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.

6.4 Clamping forces

Excessive clamping force will induce undesirable pre-loading and may damage some materials.

6.5 Friction

Most fixtures for 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.

6.6 Thin test pieces

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

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 ± 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 fixture

The fixture for the Iosipescu test is a modified asymmetric four-point bending fixture. 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. Figures 3 and 4 show a photograph and a schematic of such a fixture.

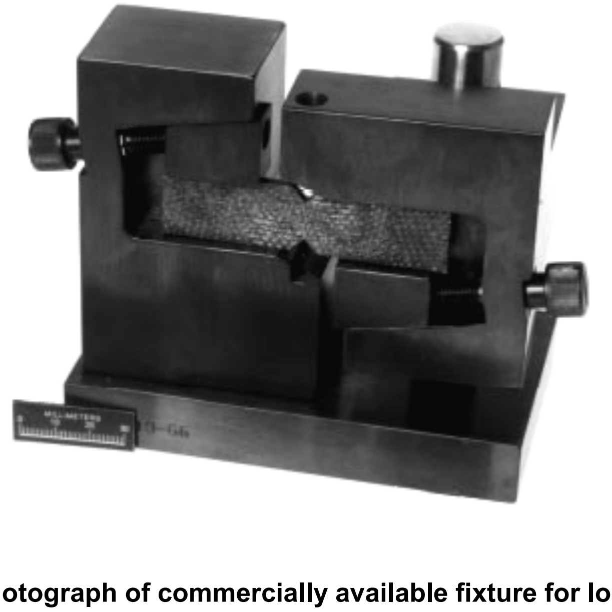
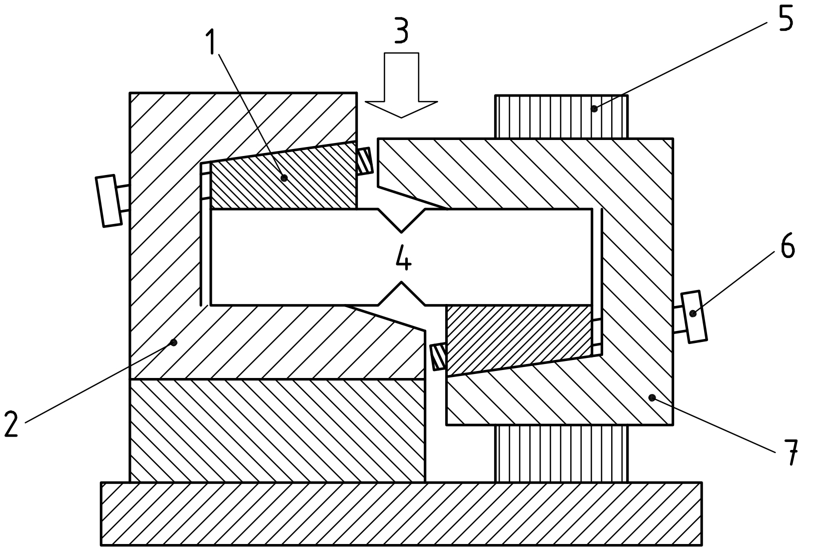


Figure 3 — Photograph of commercially available fixture for Iosipescu test



Key

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

Figure 4 — Schematic of Iosipescu test fixture

8 Test piece

8.1 Test piece geometry

The required shape and tolerances of the Iosipescu test piece are shown in Figure 2, and Table 2 contains recommended values for the dimensions of the test piece.

Table 2 — Recommended dimensions for Iosipescu test pieces

Dimension	Description	Value	Allowance
L	Test piece length	76,00 mm	± 0,1 mm
h	Distance between notches	11,00 mm	± 0,1 mm
w	Test piece width	19,00 mm	± 0,1 mm
R	Notch radius	1,30 mm	
θ	Notch angle	90,0°	
t	Test piece thickness	—	

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 procedure 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.

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 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. 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 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

Determine the thickness and notch separation in 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.

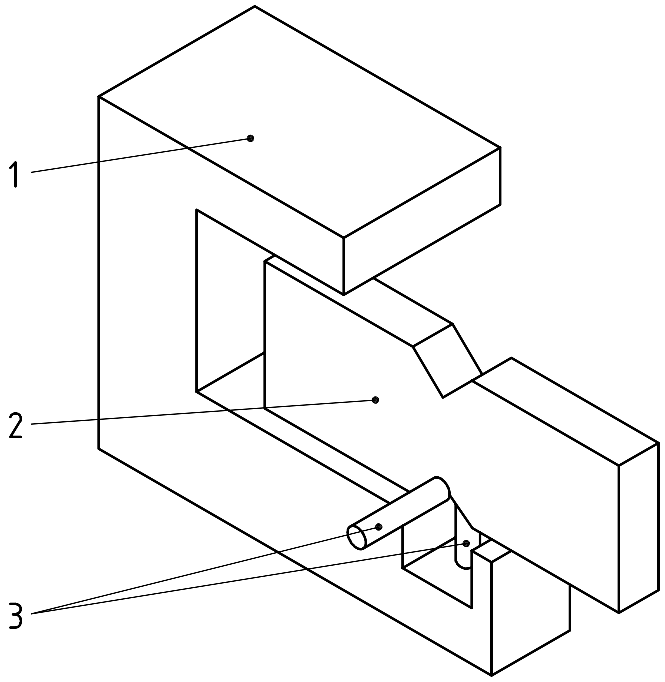
11.2 Preparations for testing

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

Loosen the jaw of each grip sufficiently to allow the test piece width to be freely inserted into the grip with clearance. Adjust the movable head position until the grips are approximately aligned vertically. Place the alignment tool in the groove in the lower fixture grip. Place the test piece loosely into both grips. Press the back side of the test piece flat against the back wall of the fixture. Pull the test piece alignment tool vertically up into the notch to centre the test piece V-notch relative to the fixture in accordance with Figure 5. While keeping the test piece centred, 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 test piece and the upper grip and no load showing in the test machine. If there is no clearance, or if load in the test piece 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 test piece 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 test piece, without loading it. Lightly tighten the jaw of the upper, right-hand, grip onto the right-hand side of the test piece. 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 test piece should now be centred in the fixture so that the line-of-action of the load acts directly through the centre of the notch on the test piece.



Key

1 fixture
2 specimen
3 alignment tool

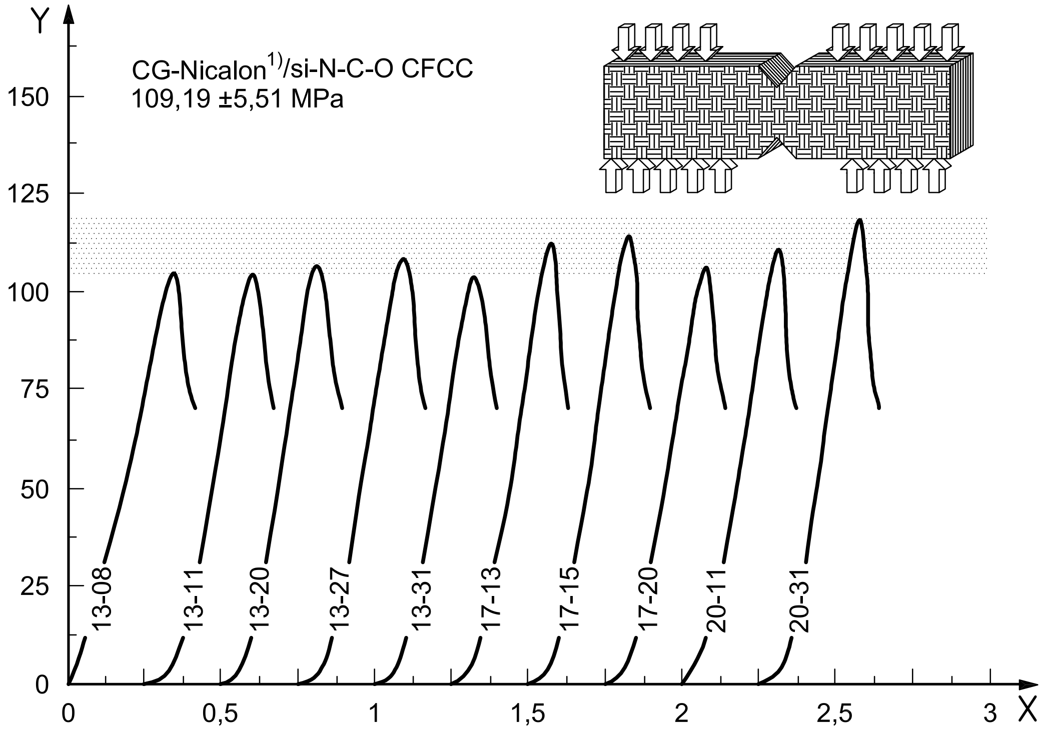
Figure 5 —Example of alignment tool

11.3.2 Begin data acquisition

11.3.3. Initiate the action of the test machine

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 ± 1 % of the load range and noted for the report. Carefully remove the test piece halves from the test piece mount. Avoid damaging the fracture surfaces by preventing them from contacting each other or other objects. Figure 6 shows typical shear stress versus displacement curves obtained for continuous-fibre-reinforced ceramic composites, while Figure 7 shows the gauge section of a Iosipescu specimen after a test.



Key

X Displacement (mm)

Y Shear strength (MPa)

Figure 6 —Typical shear stress versus displacement curves

Figure 7 — Gauge section of Iosipescu specimen after testing

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1) CG-Nicalon is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

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 a 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

Calculate the shear strength as:

$$\tau_{\mathrm{IP}} = \frac{P_{\mathrm{max}}}{A} \qquad (1)$$

where

P_{max} is the applied maximum load;

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

$$A = th \qquad (2)$$

where

t is the thickness of the test piece;

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

12.2 Statistics

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

Mean $$\overline{X} = \frac{1}{n}\sum_{i=1}^{n} X_i \qquad (3)$$

Standard deviation $$\mathrm{SD} = \sqrt{\frac{\sum_{i=1}^{n}(X_i - \overline{X})^2}{n-1}} \qquad (4)$$

Coefficient of variation $$\mathrm{CV} = \frac{100(\mathrm{SD})}{\overline{X}} \qquad (5)$$

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 shall be reported, as well as the primary processing route including green state and consolidation routes. Also report 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, 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.);

k) test mode (load or displacement control) and actual test rate (load rate or displacement rate);

l) mean, standard deviation, and coefficient of variation for the measured shear strength for each test series;

m) appearance of test piece after fracture;

n) any significant deviations from the procedures and requirements of this test method.

Annex A
(informative)

Results of round-robin tests

In 1998-1999, the US Department of Energy and the US Air Force sponsored a round-robin testing program to determine the precision and bias of ASTM Standard Test Method C1292. The repeatability and reproducibility were assessed for in-plane shear strength based on the results from the evaluation of 10 test pieces by eight laboratories. Bias was not evaluated because there is no commonly recognized standard reference material for continuous-fibre-reinforced ceramic matrix composites.

In-plane Iosipescu shear test pieces were 76 mm long, 19 mm wide, and had a nominal thickness of 3 mm. The nominal separation between the V-notches was 11 mm. The test pieces were diamond-grit cut from three panels of a commercial Sylramic S200[1] ceramic composite. The panels were fabricated with eight plies of ceramic grade Nicalon[1] fabric (8-harness satin weave) coated with boron nitride and embedded in a polymer-derived silicon-carbonitride matrix. The material had a nominal fibre volume fraction of 45 %, a mean bulk density of 2,21 g/cm^3, and average open porosity of 2,7 %.

Round-robin participants were required to perform in-plane shear strength tests in accordance with C1292. Tests were conducted in ambient conditions at a constant cross-head displacement rate of 0,05 mm/s.

A statistical analysis of the in-plane shear strength test results was performed using the procedures and criteria of Practice E691[1]. All the results for in-plane shear strength were determined to be valid and applicable. Repeatability and reproducibility are contained in Table A.1.

Sources of Variability -- The test results were analyzed for variability in experimental procedures between laboratories and for variability in materials thickness, density, and porosity among the test pieces, as well as differences between test pieces cut from the three different panels. Possible statistically significant effects were indicated for location and size of the notches with respect to the mesostructure of the material.

Table A.1 — Repeatability and reproducibility results

Mean value for the 8 laboratories	110,79 MPa	—
Standard deviation of the averages of the 8 laboratories	4,96 MPa	4,48 %
Repeatability standard deviation	2,26 MPa	2,04 %
Reproducibility standard deviation	5,39 MPa	4,88 %
95 % repeatability limit	6,33 MPa	5,71 %
95 % reproducibility limit	15,15 MPa	13,67 %

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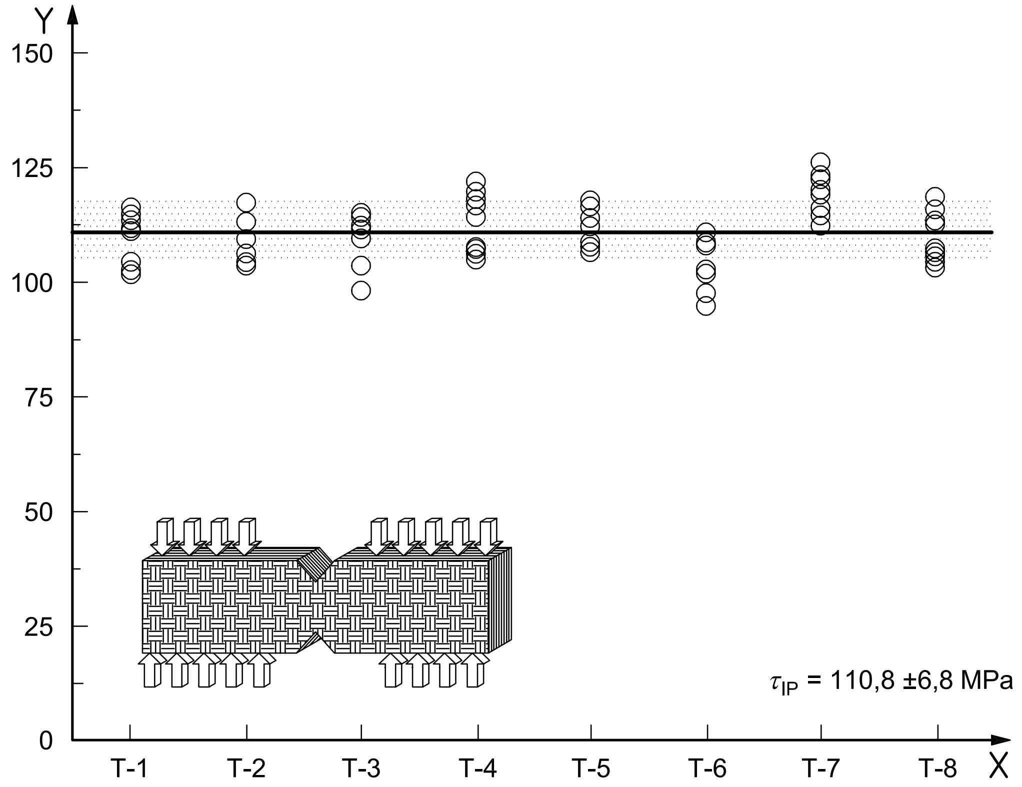
1) Sylramic S200 and Nicalon are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.



Key

Y In-plane shear strength (MPa).

Figure A.1 — Summary of results from round-robin testing programme

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[1] ASTM E691-01, *Program to Compute the Within-Lab and Between-Lab Variation of a Test Method from an Interlaboratory Round Robin Study*

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