
**Fine ceramics (advanced ceramics,
advanced technical ceramics) — Test
method for interfacial bond strength of
ceramic materials**

*Céramiques techniques — Méthode d'essai pour la résistance de
l'interface des matériaux céramiques*



Reference number
ISO 13124:2011(E)

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Foreword

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

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 13124 was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for interfacial bond strength of ceramic materials

1 Scope

This International Standard specifies a test method for determining the interfacial tensile and shear bond strength of ceramic-ceramic, ceramic-metal, and ceramic-glass joining at ambient temperature by compression tests on cross-bonded test pieces. Methods for test-piece preparation, test modes and rates (load rate or displacement rate), data collection and reporting procedures are addressed.

This International Standard applies primarily to ceramic materials, including monolithic fine ceramics and whisker-, fibre- or particulate-reinforced ceramic composites. This test method can be used for materials research, quality control, and characterization and design data generation purposes.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3611:2010, *Geometrical product specifications (GPS) — Dimensional measuring equipment: Micrometers for external measurements — Design and metrological characteristics*

ISO 7500-1:2004, *Metallic materials — Verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Verification and calibration of the force-measuring system*

ISO 14704:2008, *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.

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
cross-bonded sample

test sample in the form of a symmetrical cross, which is prepared by joining two rectangular bars with the same shape and size

NOTE 1 See Figure 1.

NOTE 2 The two bars joined to form the cross-bonded sample may be the same or different materials.

NOTE 3 The approach used for joining can be any chemical or physical bonding.

NOTE 4 The two bars should be joined perpendicularly and symmetrically within $\pm 1^\circ$ ($\alpha = 90^\circ \pm 1^\circ$).

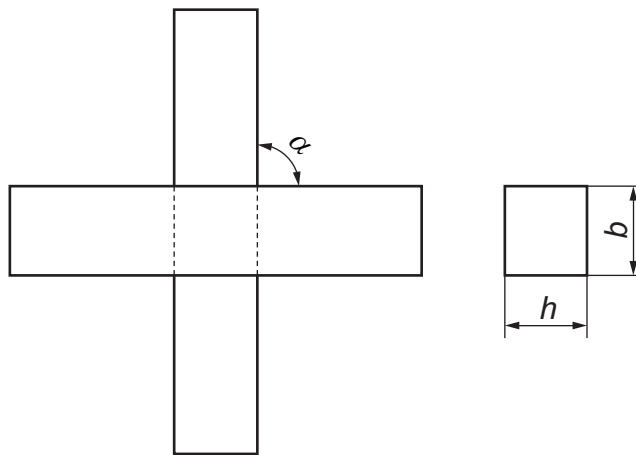


Figure 1 — Schematic diagram of the cross-bonded samples

3.3
tensile failure load

maximum tensile load applied to the interface during a tensile bond strength test

3.4
tensile bond strength

maximum mean tensile stress applied to the interface during a bond strength test

NOTE The tensile bond strength is calculated using the tensile failure load and the bonded area.

3.5
shear failure load

maximum shear load applied to the interface during a shear test of the cross-bonded sample

3.6
shear bond strength

maximum mean shear stress applied to the interface during a shear bond strength test

NOTE The shear bond strength is calculated using the shear failure load and the shear loaded area.

4 Symbols and abbreviated terms

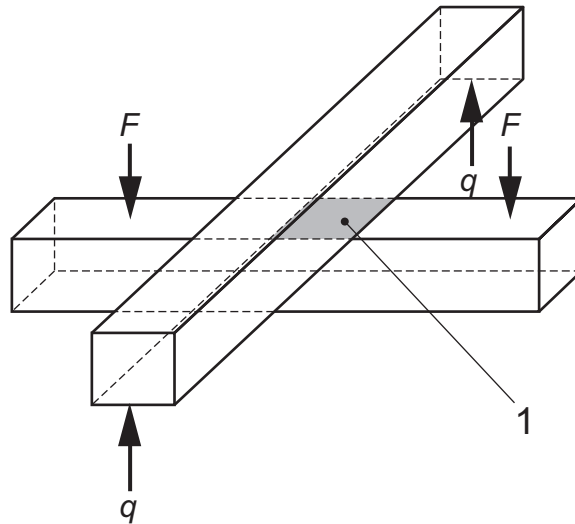
For the purposes of this document, the symbols and designations given in Table 1 apply.

Table 1 — Symbols and designations

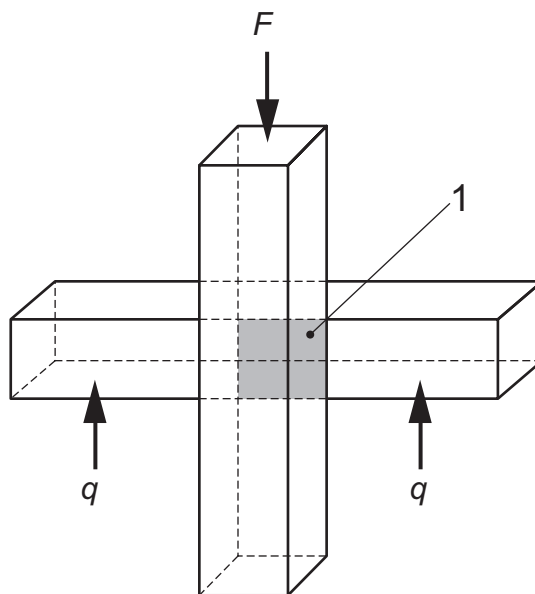
Symbol	Designation	Unit	References
l	Test-piece length	mm	Table 2
h	Test-piece thickness	mm	Figure 1, Table 2
b	Test-piece width	mm	Figure 1, Table 2
α	Right angle of cross-bonded sample	°	Figure 1
D	Diameter of the ball in pressure head	mm	Figure 3
σ_t	Tensile bond strength	MPa	Equation 1
τ	Shear bond strength	MPa	Equation 4
P_c	Critical load to debond	N	Equations 1, 4
A_1	Tensile loaded area	mm ²	Equation 1
A_2	Shear loaded area	mm ²	Equation 4
n	Number of valid tests	1	Equations 2, 3, 5, 6
$\bar{\sigma}_t$	Mean tensile bond strength	MPa	Equation 2
$\bar{\tau}$	Mean shear bond strength	MPa	Equation 5
s	Standard deviation	MPa	Equations 3, 6

5 Principle

A cross-bonded sample is loaded in compression which yields tensile or shear stress in the interface until the occurrence of debonding in the interface. Two different forms of mounting the cross-bonded sample in a fixture are designed to measure the interfacial tensile and shear bond strength, respectively. In the case of the former, a uniaxial tensile stress is generated when the testing sample is subjected to a compressive load, as shown in Figure 2 a). For the latter, a cross-bonded sample is loaded in compression to induce failure by shear at the interface, as shown in Figure 2 b). The test is usually performed at a constant cross-head displacement rate. The load at fracture and the bonded area are used to compute the tensile and shear bond strength.



a) Schematic diagram of loading, supporting and bonded area for cross-bonded sample in the test of the tensile bond strength



b) Schematic diagram of loading, supporting and bonded area for cross-bonded sample in the test of the shear bond strength

Key

- 1 loading, supporting and bonded area
- F applied load
- q uniform resultant stress on the supporting surfaces

Figure 2 — Schematic diagram of measuring the tensile and shear bond strength using the cross-bonded test piece subjected to compressive load

6 Apparatus

6.1 Testing machine

A suitable testing machine capable of applying a uniform cross-head speed shall be used. The testing machine shall be in accordance with ISO 7500-1:2004, Class 1, with an accuracy of 1 % of the indicated load during compression or tension tests.

6.2 Data acquisition

Obtain the maximum load and at least an autographic record of the applied load versus cross-head displacement or versus testing time.

Use either analog chart recorders or digital data acquisition systems. Recording devices shall be accurate to within 1 % of the selected range of the testing equipment including the readout unit, and have a minimum data acquisition rate of 10 Hz with a response of 50 Hz deemed more than sufficient.

6.3 Dimension-measuring device

Micrometers and other devices used for measuring linear dimensions must be accurate to at least 0,01 mm and shall be in accordance with ISO 3611. Alternative dimension-measuring instruments may be used provided that they have a resolution of 0,01 mm or finer.

6.4 Testing fixture

The sketch of the testing fixtures is shown in Figure 3. To avoid unsymmetrical stress in the sample, the top of the pressure head is in the centre point of the pressure head, and it is arc shaped at two perpendicular directions, as shown in Figure 3 a). Alternatively, a bearing ball inlaid in the centre of a rectangular pressure head can be used, as shown in Figure 3 b). Thus, a point-contact at the top of the pressure head can be realized in the compressive process. The supporting fixture shall be suitable and moveable, and the width of the groove such that the cross-bonded sample can be inserted into the fixture freely and with a smooth contact, as shown in Figure 3 c). The fixture shall remain elastic over the load ranges used. The fixture should be made of a hard metal with elastic modulus over 200 GPa and hardness (HV) over 3 GPa.

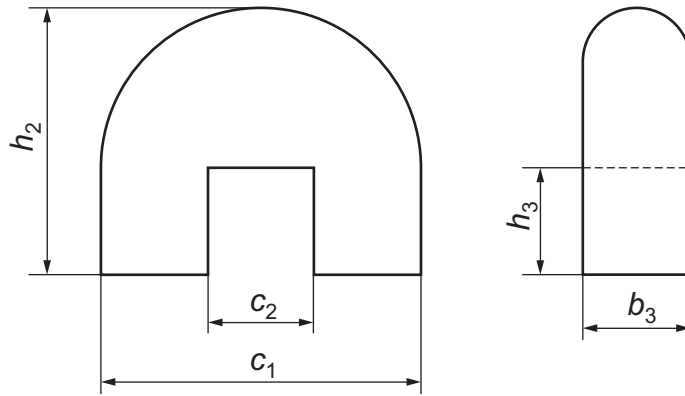
The pressure head is designed for applying the tensile load in the interface during the tensile bonding strength test, not for the shear bond strength test. The weight of the pressure head should be added into the final load for calculating the strength.

To avoid the unsymmetrical tensile stress, it is recommended that the width of the pressure head should be equal to that of the test piece, e.g. $b = b_3$.

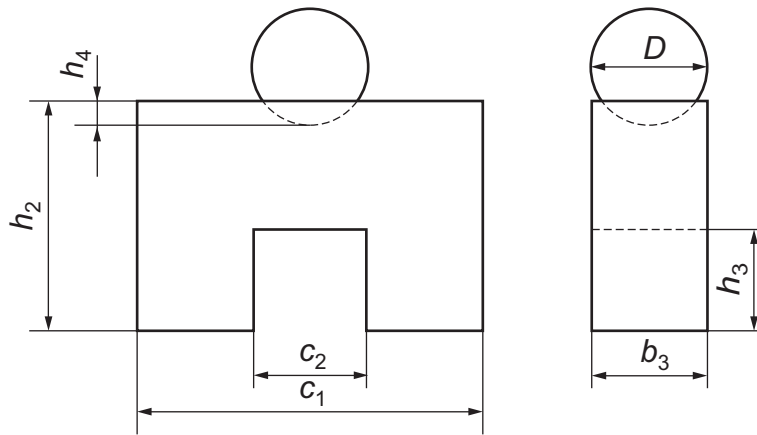
The parallelism tolerance on opposite longitudinal faces of the supporting fixture shall not exceed 0,01 mm, and both the upper and lower surfaces should be smooth planes.

NOTE While the cross-bonded sample is put into the testing fixture, as shown in Figures 5 and 6, the inside bar would be in smooth contact with two inner surfaces of the fixture, without friction when it moves.

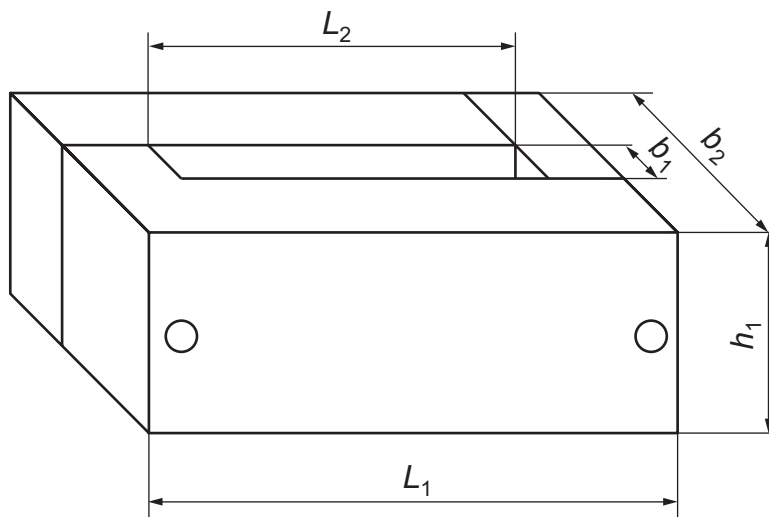
The thickness of the pressure head should be a little smaller than the width of the groove, and the depth of the groove in the pressure head is larger than the thickness of the bar, e.g. $b_3 < b_1$, $h_3 > h$.



a) Pressure head used to apply load during tensile bonding strength tests



b) Alternative pressure head used to apply load during tensile bond strength tests



c) Supporting fixture for both tensile and shear bond strength tests

Figure 3 — Schematic illustration of the testing fixtures

7 Test pieces

7.1 Test-piece size

A rectangular test bar with a square or rectangular section shall be prepared before bonding, as shown in Figure 4. The angles of the cross-section should be right-angles within $\pm 1^\circ$. Table 2 shows the recommended dimensions of the bars with the section size of 4 mm \times 4 mm, and the length should be larger than 12 mm. The parallelism tolerance on the opposite longitudinal face is 0,015 mm. All of the bar samples shall not be chamfered.

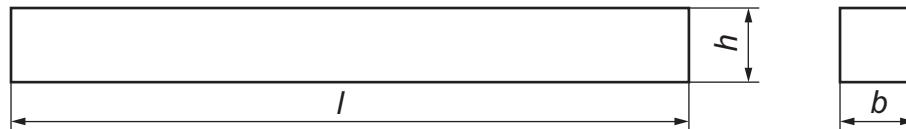


Figure 4 — Schematic illustrations of the rectangular test pieces

Table 2 — Recommended dimensions for cross-bonded sample and fixture

Dimensions in millimetres

Dimension	Description	Value	Tolerance
l	Length of the bar	>12	$\pm 0,5$
b	Width of the bar	4	$\pm 0,1$
h	Thickness of the bar	4	$\pm 0,1$

NOTE Preferably, two rectangular bars with the same dimension are joined for preparing the cross-sectional samples. The phase compositions of the two rectangular samples can be the same or different.

7.2 Test-piece preparation

7.2.1 General

This International Standard allows several options for test-piece preparation. For chemical diffusion joining, the contact surfaces of the bars are polished up to at least 1200# SiC paper, and then cleaned. For strength evaluation of glues, the surface of the test piece does not need polishing. Each pair of bars that need to bond (sometimes of the same material) is joined to form a symmetrical cross, as shown in Figure 1. The technique used for joining, depending on the aims of measurement, can be chemical or physical bonding.

7.2.2 Test-piece storage

The test pieces shall be handled with care to avoid the introduction of damage after test-piece preparation. Test pieces shall be stored separately and not allowed to impact or scratch each other.

7.2.3 Number of test pieces

A minimum of 10 test pieces is required for the purpose of estimating the mean interfacial tensile bond strength or interfacial shear bond strength.

A minimum of 30 test pieces is recommended if a statistical strength analysis (e.g. a Weibull analysis) is demanded. The use of 30 test pieces will help in obtaining good confidence limits for the bond strength distribution parameters including a Weibull modulus.

8 Test procedure

8.1 Test mode and rate

Use a universal mechanical testing machine or other appropriate fixture with a cross-head speed of 0,5 mm/min for both tensile and shear bond strength tests.

The test rate should be sufficiently rapid, so as to complete the test in the range of 10 s to 30 s, thereby obtaining the maximum possible tensile strength at fracture of the interface of the cross-bonded sample.

8.2 Preparation of test pieces

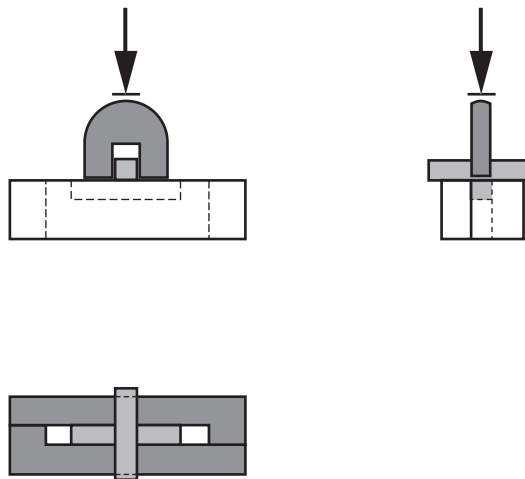
Bond two rectangular bars into a cross-bonded sample by chemical or physical means.

Check the cross-bonded sample before the test; the two bars should be rectangular to each other, and without any redundant overlap at the brim of the bonded area.

For the cross-bonded sample in shear tests, the upper section of the vertical bar might be declined in a plane in which the peak is at the side of the bond surface to avoid possible bending stress.

8.3 Measurements of the tensile bond strength

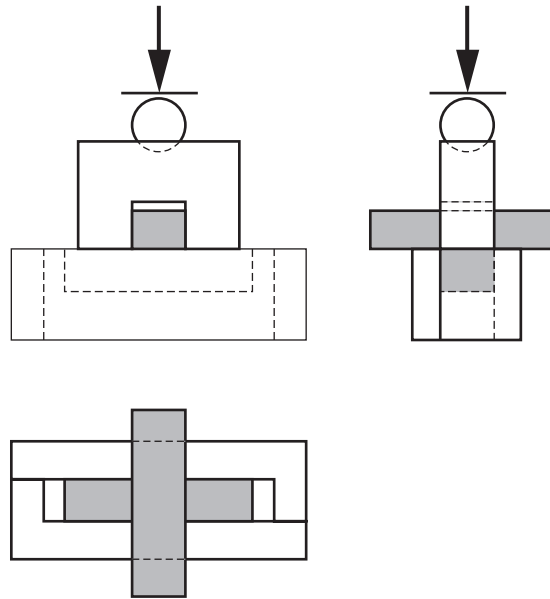
To measure the tensile bond strength, place each test piece in the fixture using an arc-shaped pressure head or flat pressure head with a ball, as shown in Figure 5. The cross-bonded sample should be inserted in the testing fixture without any friction. The lower surface of the pressure head is stuck with a soft tape for keeping uniform contact between the pressure head and the sample. The width of the pressure head must be the same as that of the bar, and the pressure head should be parallel to the lower bar. Apply the test force at the specified rate and record the peak load at fracture. Measure the peak load with an accuracy of $\pm 0,5\%$ or better.



a) Arc-shaped pressure head

Figure 5 (continued)

.....



b) Ball-shaped pressure head

Figure 5 — Schematic diagram of cross-bonded sample and fixture for measuring tensile bond strength using arc-shaped pressure head or ball-shaped pressure head

8.4 Measurements of the shear bond strength

To measure the shear bond strength, place each test piece in the fixture, as shown in Figure 6. It is recommended that a small soft tape be stuck on the upper surface of the fixture close to the side of the interface to avoid the bending moment and to keep uniform contact between the pressure head and the sample. Apply the test force at the specified rate and record the peak load at fracture. Measure the peak load with an accuracy of $\pm 1\%$ or better.

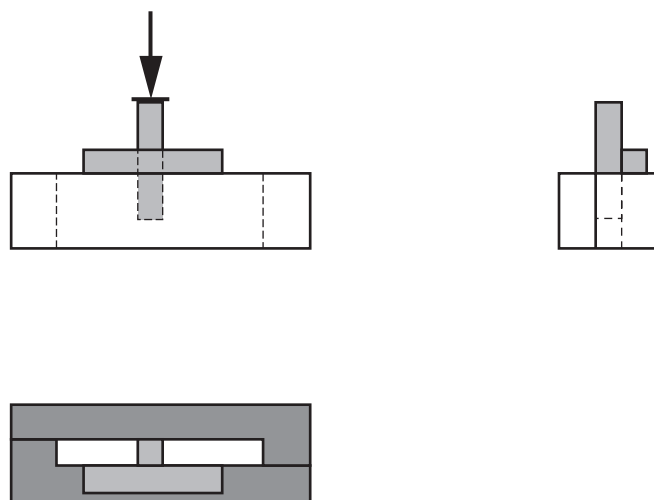


Figure 6 — Schematic diagram of cross-bonded sample and fixture for measuring shear bond strength

8.5 Measurement of the cross-bonded area

Before the bond strength test, the cross-bonded area should be measured for future calculations. Measure the length and width of the bonded area with an accuracy of 0,02 mm or better. To evaluate the quality and uniformity of the joining, the area for strength calculation can simply use the multiple of both bar-widths. Figure 7 represents the debonded sections in the tension test a) and shear test b), using the cross-bonded sample of $Ti_3SiC_2-Ti_3SiC_2$ by oxide joining.

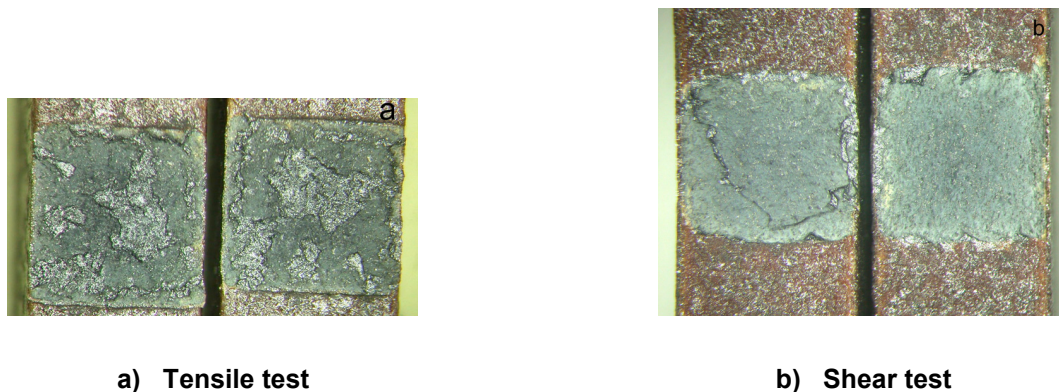


Figure 7 — Debonded sections in tensile test a) and shear test b), using the cross-bonded sample of $Ti_3SiC_2-Ti_3SiC_2$ by oxide joining

8.6 Post test

Measure and record the ambient relative humidity in the laboratory during the test sequence.

9 Calculation of results

9.1 Interfacial tensile bond strength

9.1.1 Standard formula for interfacial tensile bond strength

The standard formula for the interfacial tensile bond strength is

$$\sigma_t = \frac{P_c}{A_1} \quad (1)$$

where

σ_t is the interfacial tensile bond strength, in megapascals (MPa);

P_c is the maximum critical load to debond, in newtons (N);

A_1 is the bonded area in the tension test, in square millimetres (mm²).

9.1.2 Mean strength and standard deviation for interfacial tensile bond strength

The mean interfacial tensile bond strength, $\bar{\sigma}_t$, and the standard deviation, s , are given by:

$$\bar{\sigma}_t = \frac{\sum_{i=1}^n \sigma_{t,i}}{n} \quad (2)$$

$$s = \left[\frac{\sum_{i=1}^n (\sigma_{t,i} - \bar{\sigma}_t)^2}{n-1} \right]^{1/2} \quad (3)$$

where

$\sigma_{t,i}$ is the tensile bond strength of the i th test piece;

n is the total number of test pieces.

9.2 Interfacial shear bond strength

9.2.1 Standard formula for interfacial shear bond strength

The standard formula for the interfacial shear bond strength is:

$$\tau = \frac{P_c}{A_2} \quad (4)$$

where

τ is the interfacial shear bond strength, in megapascals (MPa);

P_c is the maximum critical load to debond under shear load, in newtons (N);

A_2 is the bonded area in the shear test, in square millimetres (mm²).

9.2.2 Mean strength and standard deviation for interfacial shear bond strength

The mean interfacial shear bond strength, $\bar{\tau}$, and the standard deviation, s , are given by:

$$\bar{\tau} = \frac{\sum_{i=1}^n \tau_i}{n} \quad (5)$$

$$s = \left[\frac{\sum_{i=1}^n (\tau_i - \bar{\tau})^2}{n-1} \right]^{1/2} \quad (6)$$

where

τ_i is the shear bond strength of the i th test piece;

n is the total number of test pieces.

10 Test report

The test report shall contain at least the following information:

- a) a reference to this International Standard;
- b) date of the test, unique identification of the report and of each page, customer name and address and signatory;
- c) name and address of the testing establishment;
- d) test specimen drawing or reference;
- e) description and test material (material type, manufacturing code, batch number);
- f) description of test specimen fabrication process; if a material process is used, report surface roughness/finish on cut or ground surface;
- g) displacement rate or deformation rate or force rate;
- h) number of tests carried out and the number of valid results obtained;
- i) valid results, mean value and standard deviations of the interfacial tensile or shear bond strength;
- j) the temperature and humidity of the laboratory.

Bibliography

- [1] BAO, Y.W., ZHANG, H.B. and ZHOU, Y.C., A simple method for measuring tensile and shear bond strength for ceramic-ceramic and metal-ceramic joining. *Materials Research Innovations*, Vol. 6 (5-6), pp. 277-280, 2002

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