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

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

Reference number
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Foreword

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ISO 17095 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 at elevated temperatures

1 Scope

This International Standard specifies the method of test for determining the interfacial tensile and shear bond strength of ceramic-ceramic, ceramic-metal, and ceramic-glass joining at elevated temperature by the compression tests on the 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 the 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 documents, in whole or in part, are normatively referenced in this document and are indispensable to its application. For dated references, only the edition cited applies. For undated references, the lasted 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 — Verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Verification and calibration of the force-measuring system*

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

3 Terms and definitions

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

3.1
advanced ceramic
advanced technical ceramic
fine ceramic
highly-engineered, high-performance predominately non-metallic, inorganic, ceramic material having specific functional attributes

3.2
cross-bonded sample
testing sample having the form of a symmetrical cross, which is prepared by joining two rectangle bars of the same shape and size

Note 1 to entry: See Figure 1.

Note 2 to entry: The two bars joined to form the cross-bonded sample may be of the same or different materials.

Note 3 to entry: The approach used for joining may be any chemical or physical bonding.

Note 4 to entry: The two bars should be joined perpendicularly and symmetrically within ± 1° (α = 90° ± 1°).

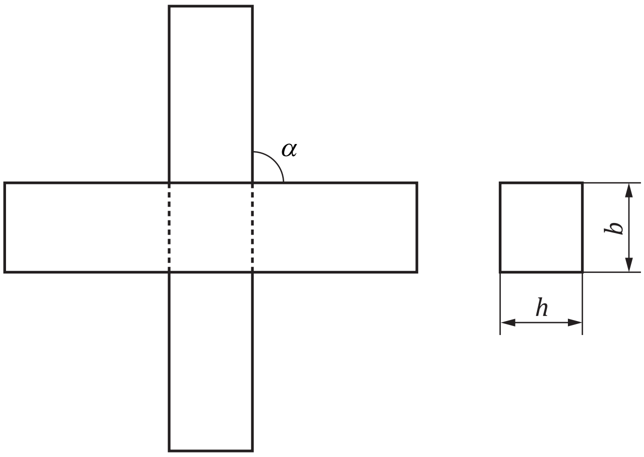


Figure 1 — Schematic diagram of cross-bonded samples

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 1 to entry: Tensile bond strength is calculated from 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 1 to entry: 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 given in Table 1 apply.

Table 1 — Symbols

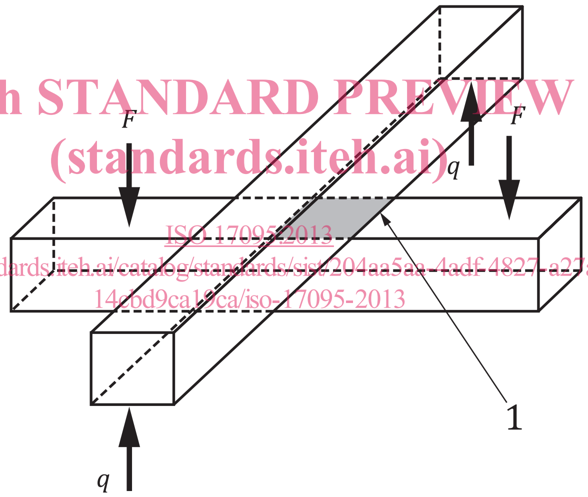
Symbol	Designation	Unit	Reference
l	Test piece length	mm	Table 2
h	Test piece thickness	mm	Figure 1, Table 2
b	Test piece width	mm	Figure 1, Table 2
α	Vertical angle of cross-bonded sample	o	Figure 1
D	Diameter of the ball in pressure head	mm	Figure 3
σ_t	Tensile bond strength	MPa	Formula (1)
τ	Shear bond strength	MPa	Formula (4)
P_c	Critical load to debonding	N	Formulae (1), (4)
A_1	Tensile loaded area	mm^2	Formula (1)
A_2	Shear loaded area	mm^2	Formula (4)

Table 1 *(continued)*

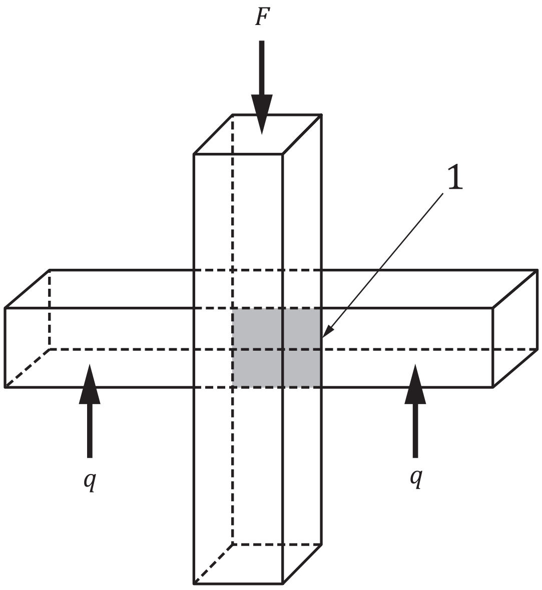
Symbol	Designation	Unit	Reference
n	Number of valid tests	1	Formulae (2), (3), (5), (6)
$\bar{\sigma}_t$	Mean tensile bond strength	MPa	Formula (2)
$\bar{\tau}$	Mean shear bond strength	MPa	Formula (5)
s	Standard deviation	MPa	Formulae (3), (6)

5 Principle

A cross-bonded sample is loaded in compression which yields tensile or shear stress in the interface until the occurrence of the debonding in the interface at the test temperatures. 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 test sample is subjected to 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 crosshead displacement rate at high temperatures. 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 tensile bond strength test



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

Key

1 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 Test machine

A suitable test machine capable of applying a uniform crosshead speed shall be used. The test machine shall be in accordance with ISO 7500-1, class 1, to an accuracy of 1 % of indicated load during compression or tension tests.

6.2 Heating machine

6.2.1 General

The furnace shall be capable of heating the test fixture and test piece as well as maintaining a uniform and constant temperature during the bonding strength test, by which an air, inert gas or vacuum environment should be available for test requirement. If an inert gas and vacuum chamber is used, and it is necessary to transmit the load through a seal, bellows or a fitting, it shall be verified that load losses or errors are less than 1 % of the expected fracture loads.

6.2.2 Test piece temperature stability

The furnace shall be controlled by a device for maintaining a constant temperature within ± 2 °C or better within the working space of the furnace, during the time that the test piece is loaded until it is fractured.

6.2.3 Test temperature uniformity

The furnace shall be capable of maintaining the test piece at uniform temperature. It shall previously be determined that the temperature of the test piece shall not vary by more than 10 °C after a 15-min hold time at the required test temperature.

6.2.4 Furnace heating rate

The furnace control device shall also be capable of controlling the heating rates of the furnace and preventing temperature overshoots.

6.2.5 Furnace stability

The time for the system to reach thermal equilibrium at the test temperature shall be determined for the test temperature to be used.

6.3 Temperature measuring and indicating instruments

6.3.1 General

The thermocouple temperature measuring equipment shall have a resolution at least 1 °C and an accuracy of 5 °C or better. Optical pyrometers, if used, shall have a resolution of at least 5 °C and an accuracy of 10 °C or better.

Note 1 Resolution is not intended to be confused with accuracy. Beware of instruments that have a resolution (readout) of 1 °C, but have an accuracy of only 10 °C; for example an instrument with a 1 % accuracy would only be accurate to ± 12 °C at 1 200 °C.

Note 2 Thermocouple temperature measuring instruments typically approximate the temperature-electromotive force (EMF) tables, but with a few degrees of error.

6.3.2 Thermocouples

Thermocouples in accordance with IEC 60584-1 shall be used. The thermocouple shall exhibit low thermal inertia (the diameter of the wires shall be not greater than 0,5 mm). The thermocouples shall have a sufficient length within the furnace (with respect to heat conduction along the wires). The measuring thermocouple tip shall be as close as possible to or contacting the test piece.

6.3.3 Verification of the thermocouple temperature measuring system

Thermocouples shall be checked periodically since calibration can drift with usage or contamination.

6.4 Data acquisition

Obtain at least an autographic record of the applied load versus crosshead displacement or testing time.

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

6.5 Dimension-measuring device

Micrometers and other devices used for measuring linear dimensions shall be accurate to at least 0,01 mm and shall be in accordance with ISO 3611. The micrometer shall not have a ball tip or sharp tip since these might damage the test piece. Alternative dimension measuring instruments may be used, provided they have a resolution of 0,01 mm or finer.