
**Fine ceramics (advanced ceramics,
advanced technical ceramics) — Test
method for determining bonding
strength of ceramic coatings**

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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.

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) — Test method for determining bonding strength of ceramic coatings

1 Scope

This document specifies the testing method for the determination of the bonding strength of ceramic coatings at ambient temperature by the compression tests on the cross-joined test pieces. Methods for test piece preparation, test mode and rate, data collection and reporting procedures are addressed.

This document applies primarily to any ceramic coatings, thick or thin, bonded onto substrates of various materials. The test method described can be used for materials research, quality control, characterization and design data generation purposes.

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*

3 Terms and definitions

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

ceramic coating

coating of ceramics onto a substrate, via a physical or chemical technique, which creates an interface or boundary between the coating and the substrate

3.2

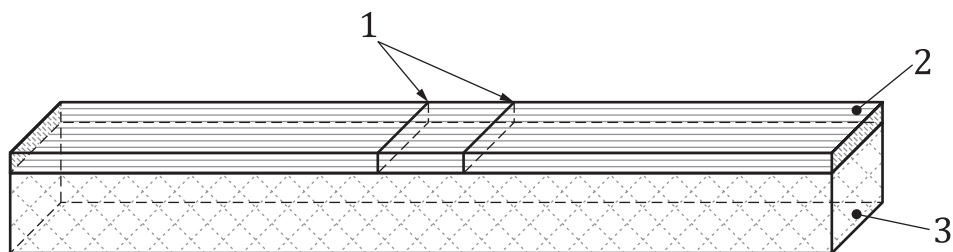
interface

smooth or rough boundary generated between coating and substrate that results from one or several bonding mechanisms, for example mechanical anchorage, interatomic or intermolecular bonds

3.3

cross-joined test piece

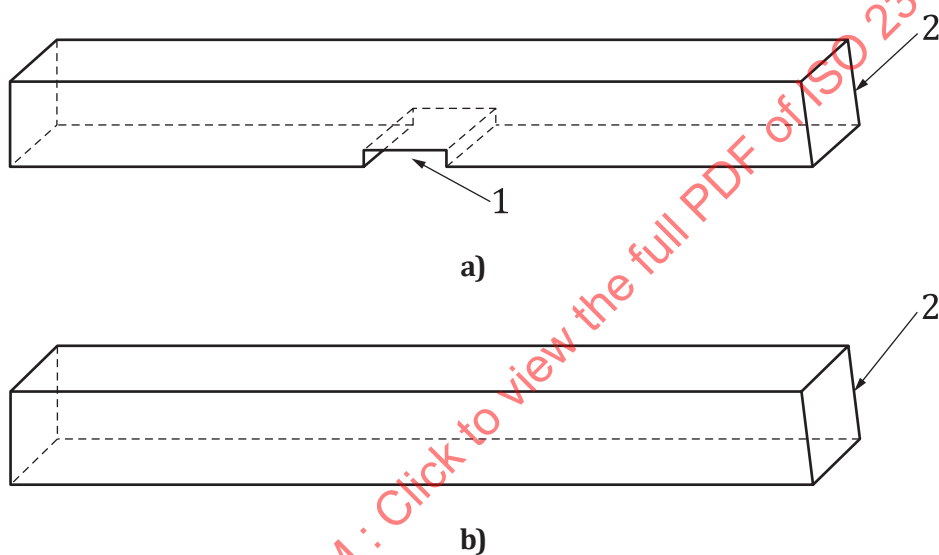
test piece prepared by joining coating sample (see [Figure 1](#) and [7.1.1](#)) and coupling bar (see [Figure 2](#) and [7.2.2](#)) perpendicularly to each other at middle, by means of high-strength adhesive, as shown in [Figure 3](#)



Key

- 1 notch
- 2 coating
- 3 substrate

Figure 1 — Schematic of the coating sample



Key

- 1 groove
- 2 sloped end

Figure 2 — Schematic of the coupling bar

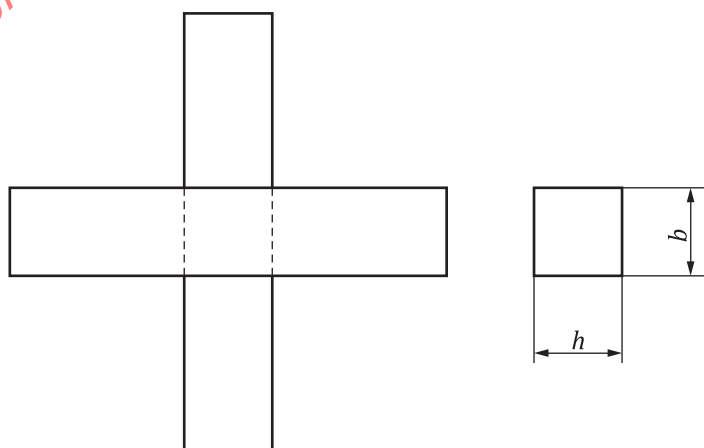


Figure 3 — Schematic of the cross-joined test piece

3.4**tensile failure load**

maximum load applied to the horizontally placed cross-joined test piece during a test by subjecting load normal to interface between coating and substrate

3.5**tensile bonding strength**

tensile stress calculated from the tensile failure load and the cross-joined area

3.6**shear failure load**

maximum load applied to the vertically placed cross-joined test piece during a test by subjecting load parallel to interface between coating and substrate

3.7**shear bonding strength**

shear stress calculated by using the shear failure load and the cross-joined area

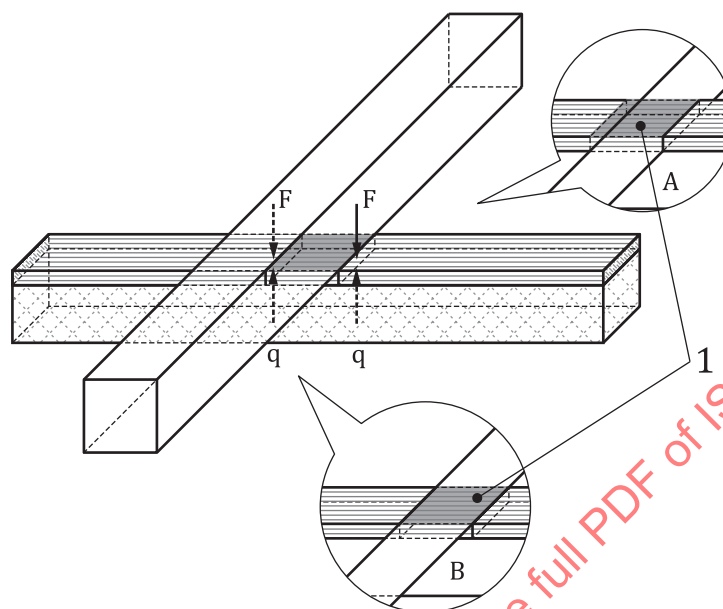
4 Symbols

Symbol	Designation	Unit	References
A_0	Cross-joined area ($=b^2$)	mm ²	Formulae (1) and (2)
A_c	True coating debonded area	mm ²	8.5
b	Test piece width	mm	Table 1
b_1	Groove width of sample holder	mm	Figure 6 b)
b_2	Sample holder width	mm	Figure 6 b)
b_3	Pressure head width ($=b$)	mm	Figure 6 a)
c_1	Pressure head length ($<l_2$)	mm	Figure 6 a)
c_2	Slot span of pressure head ($>b$)	mm	Figure 6 a)
d	Space of two notches ($=b$)	mm	Table 1
h	Test piece thickness	mm	Table 1
h_1	Sample holder height	mm	Figure 6 b)
h_2	Pressure head height	mm	Figure 6 a)
h_3	Slot depth of pressure head ($>b$)	mm	Figure 6 a)
l	Test piece length	mm	Table 1
l_1	Sample holder length	mm	Figure 6 b)
l_2	Groove length of sample holder ($>c_1$)	mm	Figure 6 a)
P_c	Failure load to debonding	N	Formulae (1) and (2)
σ_t	Tensile bonding strength by calculation	MPa	Formula (1)
σ_{ct}	Tensile bonding strength of ceramics coating	MPa	9.1
τ	Shear bonding strength by calculation	MPa	Formula (2)
τ_c	Shear bonding strength of ceramics coating	MPa	9.2

5 Principle

A cross-joined test piece is subjected to an increasing compression load giving a tensile or shear stress on the area that is joined until the coating is debonded. Two different forms of mounting the cross-joined test piece in a fixture are conducted to measure the tensile and shear bond strength, respectively. In the case of the tensile test, a uniaxial tensile stress is generated when the cross-joined test piece is subjected to compressive load, as shown in [Figure 4](#). The tensile strength of the coating–substrate interface, or the cohesive strength of the ceramic coating, will be obtained. In the case of the shear strength test, a cross-joined test piece is loaded in compression to induce failure by shear at the interface, as shown in

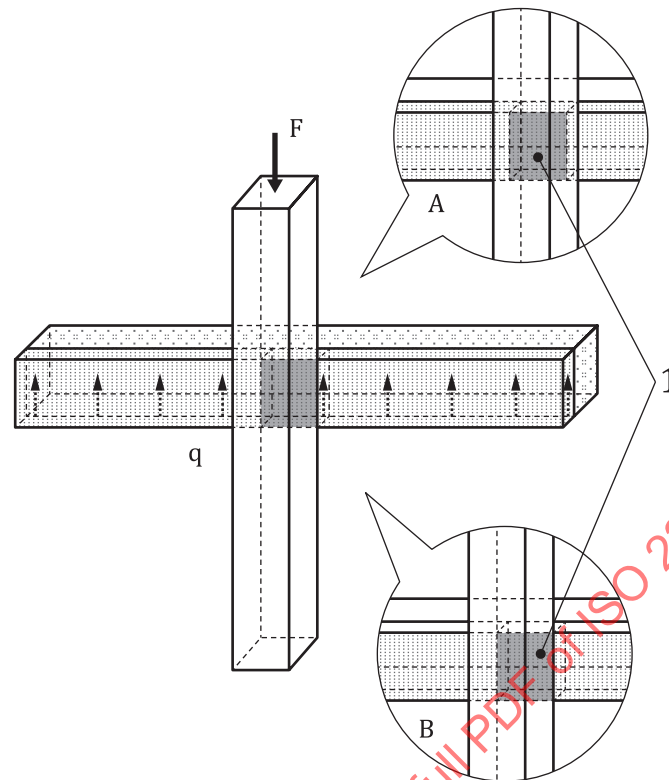
Figure 5. Owing to the groove, the failure will not occur in the adhesive interface, and thus the shear bonding strength shall be that of ceramic coating to be characterized. On the other hand, if that coating sample is joined with a flat coupling bar, the adhesive may also lead to the removal of the coating from substrate under shear stress. The test is performed at a constant cross-head displacement rate. The load at fracture and the cross-joined area are used to calculate the tensile and shear bonding strengths by calculation.



Key

- F loading
- q supporting
- 1 jointed area (dark)
- A grooved coupling bar
- B flat coupling bar

Figure 4 — Schematic of loading, supporting and jointed area (dark) for cross-joined test piece in the test of the tensile bonding strength



Key

- F loading
- q supporting
- 1 joined area (dark)
- A grooved coupling bar
- B flat coupling bar

Figure 5 — Schematic of loading, supporting and jointed area (dark) for cross-joined test piece in the test of the shear bonding strength

There are three possible fracture modes during either tensile or shear testing: a) complete removal of the coating from substrate (debonded at the interface between coating and substrate); b) the coating itself fractures (cohesive failure inside the coating); and c) adhesive joint fails. A combination of these three modes can occur. For mode a), the calculated strength value represents the real bonding strength of the coating. For mode b) and mode c), the strength calculated with the failure load and the jointed area shall be lower than the real bonding strength between the coating and the substrate.

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, Class 1, with an accuracy of 1 % of indicated load at compression or tension tests.

6.2 Data acquisition

Obtain an autographic record of the applied load versus cross-head displacement or versus testing time, so that the maximum load to debonding (P_c) is determined.

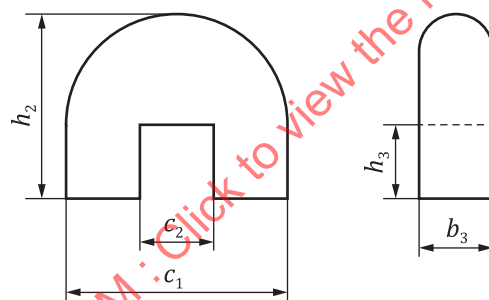
Use either analogue chart recorders or digital data acquisition systems. Recording devices shall be accurate to within 1 % of the selected range of the testing equipment, including readout unit, and shall have a minimum data acquisition rate of 10 Hz with a response of 50 Hz deemed more than sufficient.

6.3 Dimension-measuring device

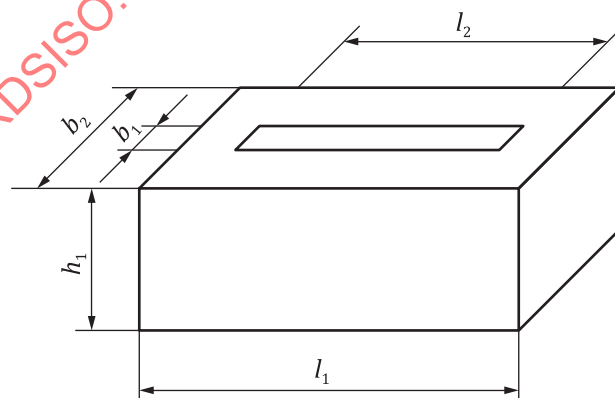
Micrometers and other devices used for measuring linear dimensions shall be accurate to at least 0,01 mm in accordance with ISO 3611. The micrometer shall not have a ball tip or sharp tip since these might damage the test piece. Alternative dimension-measuring instruments may be used provided that they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

6.4 Testing fixture

Preferred testing fixtures (pressure head and sample holder) are shown in Figure 6. To avoid unsymmetrical stress in the sample, the top of the pressure head shall be machined into an arc shape in two perpendicular directions, as shown in Figure 6 a). Thus, a point contact at the top centre of the pressure head can be realized in the compressive process. The slot of pressure head shall be slightly bigger than the cross-section of the coupling bar, i.e. $c_2 > b$, $h_3 > b$ [see Figure 6 a) and Figure 8], to leave adequate clearance between the two. The groove of sample holder shall be suitable in width (b_1) to create a smooth contact between its two inner surfaces and inserted cross joined test piece, ensuring the coating sample (tensile bonding strength test) or coupling bar (shear bonding strength test) can move freely during testing, as shown in Figure 6 b). The fixture shall remain elastic over the load ranges used. The fixture shall be made of a hard material, such as steel, or a ceramic with an elastic modulus over 200 GPa.



a) The pressure head used to apply the load during the tensile bonding strength tests



b) The sample holder for both tensile and shear bonding strength tests

Figure 6 — Schematic illustration of the testing fixtures

The pressure head is designed to apply the tensile load during the tensile bonding strength tests, which is unnecessary for measuring the shear bond strength. The weight of the pressure head shall be added into the final load for calculating the strength.

To avoid unsymmetrical tensile stress, it is recommended that the width of the pressure head be equal to that of the testing sample, i.e. $b = b_3$.

The parallelism tolerance on opposite longitudinal faces of the lower part of the fixture (holder) shall not exceed 0,01 mm, and the upper surface shall be a smooth plane.

When the cross-joined test piece is put into the testing fixture, as shown in [Figure 8](#) and [Figure 9](#), the inside bar shall be in smooth contact with two inner surfaces of the holder, without friction when it moves.

7 Test pieces

7.1 Test piece preparation

7.1.1 Coating sample

A coating sample shall be machined into a rectangular bar up to recommended dimensions of 4 mm × 4 mm × 20 mm, as indicated in [Figure 1](#), [Figure 7](#) and [Table 1](#). Two notches shall then be sliced in the middle to separate the testing area, as shown in [Figure 1](#). The space between two notches shall equal the width of the coupling bar (see [7.1.2](#) and [Table 1](#)). The parallelism tolerance on opposite longitudinal faces shall be 0,015 mm. None of the sample bars shall be chamfered.

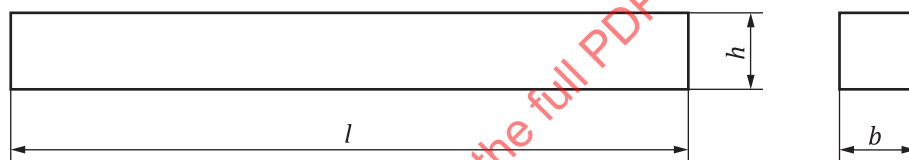


Figure 7 — Schematic illustration of the rectangular coating sample

Table 1 — Recommended dimensions for coating sample

Dimension	Description	Value mm	Tolerance mm
l	Length	20	$\pm 0,5$
b	Width	4	$\pm 0,1$
h	Thickness	4	$\pm 0,1$

7.1.2 Coupling bar

A coupling bar of the same size as the coating sample shall be made from typical stainless steel and a small slope shall be ground on one end. A groove, of less depth than the ceramic coating, shall be sliced to match the middle testing area of the coating sample, as shown in [Figure 2 a\)](#), or for thin coatings the coupling bar without groove may also be used [see [Figure 2 b\)](#)].

7.1.3 Joining of coating sample with coupling bar

Before joining, the surface of the groove or joining area on the coupling bar shall be polished up to at least 1200# SiC paper and then cleaned. The coating sample, especially for the testing area, shall also be cleaned.

The coating sample and coupling bar shall be mounted by matching the testing area of the coating sample (between two notches) with the groove of the coupling bar (or with the flat bar in the middle), and both shall be joined by using high-strength adhesive to form a cross-joined test piece, as indicated in [Figure 3](#) and [3.3](#).

The approach used for joining shall ensure that the joining does not harm the coating or that any reaction occurs between adhesive and coating.

7.2 Test piece storage

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

7.3 Number of test pieces

A minimum of 10 test pieces are required to give an overall estimation of tensile bond strength or shear bond strength of coating.

8 Test procedure

8.1 Placement of testing pieces

Check the cross-joined test piece before the test. The coating and coupling bars should be perpendicular to each other.

For the cross-joined test piece in shear tests, the coupling bar with a sloped end (see [Figure 3](#)) shall be placed above with the peak at the side of the join surface to avoid possible bending stress, as shown in [Figure 5](#).

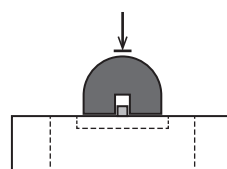
8.2 Test mode and rate

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

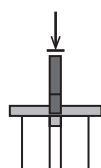
The test rate shall be sufficiently rapid to complete the test in the range of 10s to 30s, thereby obtaining the maximum possible tensile strength for coating debonding from substrate.

8.3 Measurements of the tensile bonding strength

To measure the tensile bonding strength, place each test piece in the fixture, as shown in [Figure 8](#). The cross-joined test piece shall be inserted in the testing fixture without any friction. The lower surface of the pressure head shall be stuck with a soft tape to maintain uniform contact between the pressure head and the sample. The width of the pressure head shall be the same as that of the bar, and the pressure head shall be parallel to the lower bar, as shown in [Figure 8 b\)](#). 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.



a)



b)

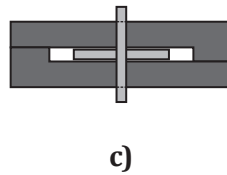


Figure 8 — Schematic diagram of cross-joined test piece and fixture for measuring tensile bonding strength

8.4 Measurements of the shear bonding strength

To measure the shear bonding strength, place each test piece in the fixture, as shown in [Figure 9](#). The soft tape shall be also stuck on the upper surface of the fixture for keeping 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.

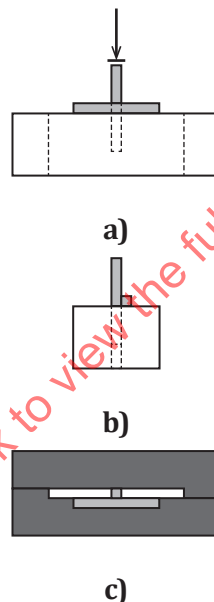


Figure 9 — Schematic diagram of cross-joined test piece and fixture for measuring shear bonding strength

8.5 Evaluation of the true coating debonded area

Apply an optical micrograph to evaluate the true area of coating debonded from the substrate (A_c) after fracture to confirm its percentage of the cross-joined area (A_0). 50 % is defined as the critical value. A photomicrograph of the coating debond area shall be taken to reveal the fracture mode.

8.6 Post test

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

9 Calculation of results

9.1 Tensile bonding strength of ceramics coating

Tensile bonding strength by calculation shall be derived from [Formula \(1\)](#).

$$\sigma_t = \frac{P_c}{A_0} \quad (1)$$

where

σ_t is the tensile bonding strength by calculation, in megapascals (MPa);

P_c is the failure load to debonding, in newtons (N);

A_0 is the cross-joined area in tensile test, $A_0 = b^2$, in mm².

If the true coating debonded area (A_c) is larger than 50 % of the A_0 , then the tensile bonding strength of coating (σ_{ct}) shall be considered equal to σ_t . Otherwise, the tensile bonding strength of coating (σ_{ct}) shall be qualitatively determined as $\sigma_{ct} > \sigma_t$.

9.2 Shear bonding strength of ceramics coating

Shear bonding strength by calculation shall be derived from [Formula \(2\)](#).

$$\tau = \frac{P_c}{A_0} \quad (2)$$

where

τ is the shear bonding strength by calculation, in megapascals (MPa);

P_c is the failure load to debonding, in newtons (N);

A_0 is the cross-joined area in shear test, $A_0 = b^2$, in mm².

If the true coating debonded area (A_c) is larger than 50 % of the A_0 , then the shear bonding strength of coating (τ_c) shall be considered equal to τ . Otherwise, the shear bonding strength of coating (τ_c) shall be qualitatively determined as $\tau_c > \tau$.

10 Analysis of precision and uncertainty

The precision of the interfacial strength measurement of ceramic coatings may be affected by many factors, for example the uniformity of the interface joining, the effects of the loading and changes in the test conditions.

Since fracture has three possible modes, the test results are either quantitative or qualitative, as mentioned in [Clause 5](#). Only for mode a) is the calculated strength the real interfacial strength. The analysis of uncertainty for mode a) fracture is given in [Annex A](#), through interlaboratory testing.

11 Test report

The test report shall contain at least the following information:

- a) name and address of the testing establishment;