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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for interfacial tensile and shear fatigue properties of ceramic joining loaded in constant amplitude at room temperature

Céramiques fines (céramiques avancées, céramiques techniques avancées) — Méthode d'essai relatives aux propriétés de tension interfaciale et de fatigue en cisaillement des jonctions céramiques à amplitude constante

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Foreword

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

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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for interfacial tensile and shear fatigue properties of ceramic joining loaded in constant amplitude at room temperature

1 Scope

This document specifies a test method for determining interfacial tensile/tensile or shear/shear cyclic fatigue properties of ceramic-ceramic, ceramic-metal, and ceramic-glass joining loaded in the constant amplitude at room temperature. Procedures for test piece preparation, test modes and rates (load rate or displacement), data collection and reporting procedures are given.

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

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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*

ISO 13124, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for interfacial bond strength of ceramic materials*

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 http://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
cross-bonded test piece
test sample in the form of a symmetrical cross

Note 1 to entry: Prepared by joining two rectangular bars with the same shape and size, as described in ISO 13124.

3.2
average applied stress during cyclic fatigue
σ_m
average value between the maximum applied stress and the minimum applied stress during cyclic fatigue

3.3
constant amplitude loading
under a given average applied stress, σ_m, keeping peak loads and valley loads constant for wave form loading in cyclic fatigue loading, $\Delta\sigma = (\sigma_{max} - \sigma_{min})/2 = \sigma_{max} - \sigma_m = \sigma_m - \sigma_{min}$

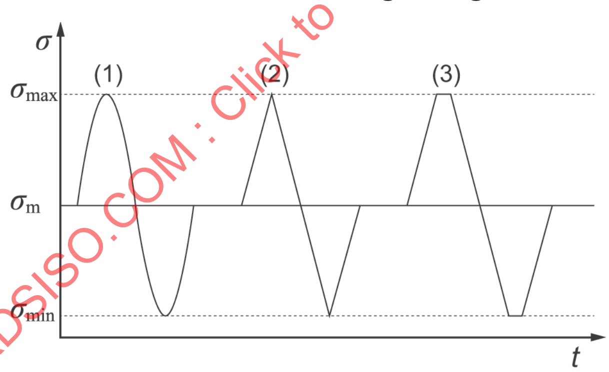
3.4
number of cycles
N
total number of loading cycles applied to the test piece during the test

3.5
cyclic fatigue life
N_f
total number of loading cycles until the test piece is up to failure

3.6
time to failure
t_f
time duration until the test piece is up to failure

4 Principle

A cross-bonded test piece is cycled under a given average stress with constant amplitude loading at room temperature, which yields cyclic tensile/tensile or shear/shear stress in the interface. Typical cyclic loading waves including sine wave, triangular wave and trapezoidal wave are shown in Figure 1. Two different forms of mounting the cross-bonded test piece in a fixture are designed to measure the interfacial tensile/tensile and shear/shear fatigue properties by using compressive/compressive fatigue loads, respectively. The total number of cycles is recorded. The lifetime duration including cyclic fatigue time and time to failure or the residual interfacial bonding strength is determined.



Key

1 sine wave
2 triangular wave
3 trapezoidal wave
σ applied stress
σ_{max} maximum applied stress during cyclic fatigue
σ_{min} minimum applied stress during cyclic fatigue
σ_m average applied stress during cyclic fatigue, $\sigma_m = (\sigma_{max} + \sigma_{min})/2$
t testing time

Figure 1 — Schematic illustration of three typical cyclic loading waves

NOTE 1 The typical fatigue test is defined by cyclic loading, average stress, constant amplitude, environment and frequency. The loading condition has the form $\sigma = \sigma_m \pm \Delta\sigma$ under a given frequency.

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NOTE 2 The complete lifetime diagram is expensive and time consuming because of the use of a great number of test pieces. It is sufficient to know the cyclic fatigue under specified stress conditions or to measure the fatigue limit.

5 Apparatus

5.1 Fatigue test machine.

A hydraulic type or electric actuator driven test machine with load control operated mode shall be used. The system for measuring the force applied to the test specimen shall be designed for fatigue tests and shall conform with ISO 7500-1. The machine shall be equipped with a cycle counter for the chosen test frequency.

5.2 Data recording system.

A calibrated recorder shall be used to record a force-time curve. A digital data recording system is recommended.

5.3 Micrometers.

Micrometers used for measuring the specimen dimensions shall conform to ISO 3611.

5.4 Testing fixture.

The sketch of the testing fixtures is shown in Figure 2. To avoid unsymmetrical stresses in the interface of the testing piece, the top of the pressure head is in the symmetrical axis of the pressure head, and it is arc shaped at two perpendicular directions, as shown in Figure 2 a). 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 test piece can be inserted into the fixture freely and with a smooth contact, as shown in Figure 2 b). The fixture shall remain elastic over the load ranges used. The fixture should be made of hard metal with elastic modulus over 200 GPa and hardness (HV) over 3 GPa.

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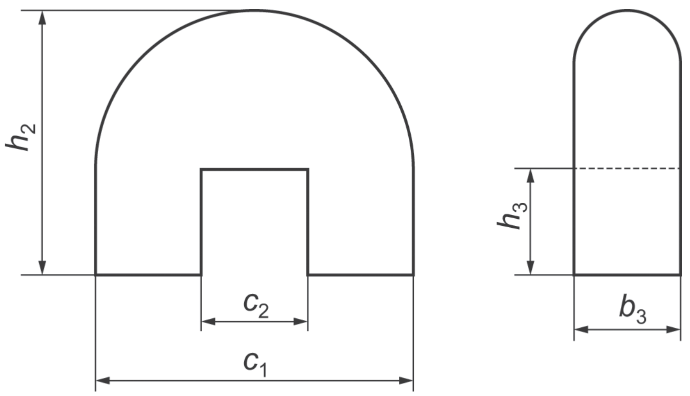
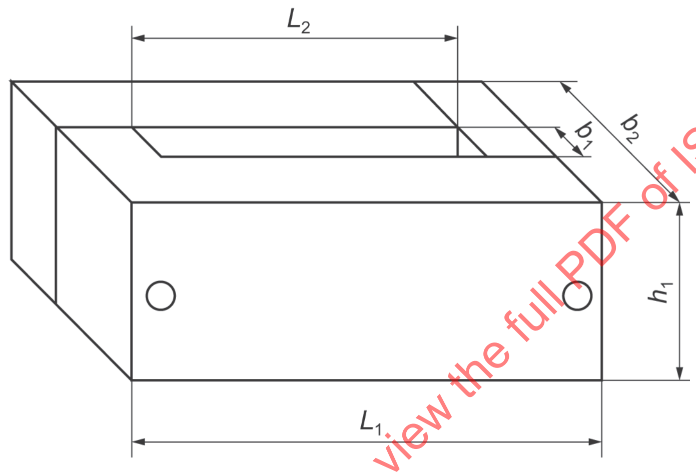


a) Pressure head used to apply cyclic load during interfacial tensile fatigue properties tests



b) Supporting fixture for both interfacial tensile and shear fatigue properties tests

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Key

C_1 outer length of the pressure head

C_2 inner length of the pressure head

L_1 outer length of the supporting fixture

L_2 inner length of the supporting fixture

h_1 height of the supporting fixture

h_2 height of the pressure head

h_3 depth of the groove in the pressure head

b_1 inner width of the supporting fixture

b_2 outer width of the supporting fixture

b_3 width of the pressure head

Figure 2 — Schematic illustration of the testing fixture

The pressure head is designed for applying the tensile load in the interface during the tensile fatigue properties test, not for the shear fatigue properties test. The weight of the pressure head should be added to the value of the final applied load for calculating the interfacial tensile stress.

To avoid the presence of unsymmetrical tensile stresses, the width of the pressure head shall be equal to that of the test piece, i.e. $b = b_3$. 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 test piece is put into the testing fixture, the inside bar would be in smooth contact with two inner surfaces of the fixture, without friction when it moves.

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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, i.e. $b_3 < b_1$, $h_3 > h$.

6 Test pieces

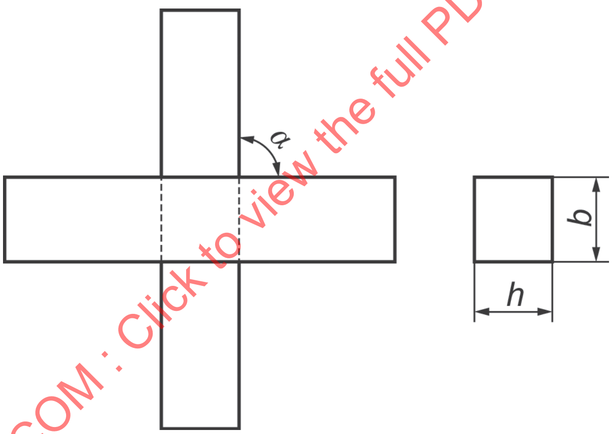
6.1 Test-piece size

Rectangular test pieces with square or rectangular sections shall be prepared before bonding. The angles of the cross-sections should be right-angles within ±1°. The recommended dimensions of the bars are 4 mm × 4 mm × 12 mm. The parallelism tolerance on the opposite longitudinal face is within 0,015 mm. The bar test pieces shall not be chamfered.

6.2 Test-piece preparation

6.2.1 General

The technique used for joining the bars, depending on the aims of measurement, can be chemical or physical bonding. For chemical diffusion joining, the contact surfaces of the bars are polished up to at least 1200# SiC paper and then cleaned. Each pair of bars that need to bond (sometimes of the same material) is joined to form a symmetrical cross (Figure 3), as described in ISO 13124.



Key

α angles between two bars

b width of the test piece

h height of the test piece

Figure 3 — Schematic diagram of the cross-bonded test piece

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6.2.2 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.

6.2.3 Number of test pieces

A minimum of three valid test results are required for any condition.

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7 Test procedure

7.1 Measurement of the test piece dimensions

The interfacial bonding area shall be determined before fatigue test. Dimensions of test piece size and the bonding area size shall be measured to an accuracy of 0,02 mm. To evaluate the quality and uniformity of the joining, the area for strength calculation can simply use the multiple of both bar-widths. The arithmetic means of the measurements shall be used for calculations.

7.2 Measurement of the interfacial tensile fatigue properties

To measure the tensile fatigue properties, put each test piece in the fixture using an arc-shaped pressure head, as shown in Figure 4. The cross-bonded test piece 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 test piece. The width of the pressure head shall 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 frequency and record the total cyclic number.

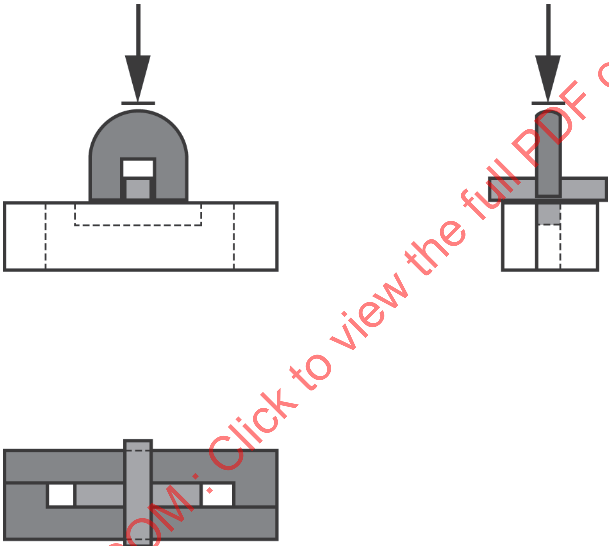

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Figure 4 — Schematic diagram of the cross-bonded test piece and fixture for measuring the interfacial tensile/tensile fatigue properties

Measurements:

a) zero the load cell;

b) set the maximum number of cycles, N;

c) set the minimum force value, usually 2 N to 5 N recommended;

d) set the maximum force value, usually 70 % to 90 % of the fracture load recommended;

e) set the frequency and the wave shape;

f) start the fatigue test in load control mode;

g) record the number of cycles, N, or cyclic fatigue life, N_f.

7.3 Measurement of the interfacial shear fatigue properties

To measure the shear fatigue properties, place each test piece in the fixture, as shown in Figure 5. It is recommended that a small soft tape be stuck on the upper surface of the fixture close to the side of the

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interface to avoid the bending moment and to keep uniform contact between the pressure head and the test piece. Apply the test force at the specified frequency and record the total cyclic number.

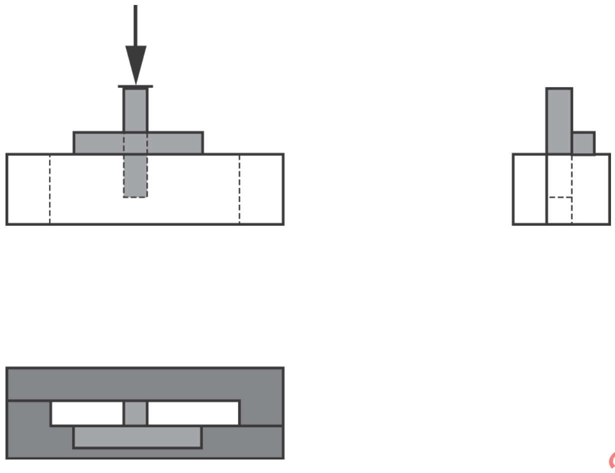

Figure 5 — Schematic diagram of the cross-bonded test piece and fixture for measuring the interfacial shear/shear fatigue properties

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Measurements:

a) zero the load cell;

b) set the maximum number of cycles, N;

c) set the minimum force value, usually 2 to 5 N recommended;

d) set the maximum force value, usually 70 % to 90 % of the fracture load recommended;

e) set the frequency and the wave shape;

f) start the fatigue test in load control mode;

g) record the number of cycles, N, or cyclic fatigue life, N_f.

The maximum load can be also given according to service requirement of a component. A specified computer program is recommended to control the test. Depending on the computerized facilities used, all the loops can be recorded. If this is not possible, the following sequence can be used to record stress versus testing time:

1) every cycle for the first 10 cycles;

2) one every 10 cycles between 10 and 100 cycles;

3) one every 100 cycles between 100 and 1000 cycles;

4) one every 1 000 cycles between 1 000 and 10 000 cycles, etc.

7.4 Residual properties

When rupture does not occur before the end of the test, the residual interfacial tensile or shear bonding strength at room temperature shall be tested in accordance with ISO 13124.

7.5 Test validity

The following circumstances shall invalidate a test for the determination of the lifetime duration:

— failure to specify and record test conditions;

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