BS ISO 19603:2016

BSI Standards Publication

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for determining elastic modulus and bending strength of thick ceramic coatings

National foreword

This British Standard is the UK implementation of ISO 19603:2016.

The UK participation in its preparation was entrusted to Technical Committee RPI/13, Advanced technical ceramics.

A list of organizations represented on this committee can be obtained on request to its secretary.

Published by BSI Standards Limited 2016

ISBN 978 0 580 86513 8

ICS 81.060.30

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 November 2016.

Amendments/corrigenda issued since publication

Date	Text affected

INTERNATIONAL
STANDARD

ISO
19603

First edition
2016-11-01

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for determining elastic modulus and bending strength of thick ceramic coatings

Céramiques techniques — Méthode d'essai relative à la détermination du module élastique et de la résistance en flexion des revêtements de céramique épais

Reference number
ISO 19603:2016(E)

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Foreword

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The committee responsible for this document is ISO/TC 206, *Fine ceramics*.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for determining elastic modulus and bending strength of thick ceramic coatings

1 Scope

This document specifies a testing method for determining the elastic modulus and bending strength of thick ceramic coatings at ambient temperature by three-point bending tests. Procedures for test piece preparation, test modes and load rates, data collection and reporting are given.

This document applies to thick, brittle coatings on metal or ceramic substrates. This test method can be used for material 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:

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

3.1
elastic modulus
ratio of stress to strain

Note 1 to entry: Also known as Young's modulus.

3.2
bending strength
maximum tensile stress at fracture under bending load

3.3
modulus ratio
ratio of the coating modulus to the substrate modulus

3.4
thickness ratio
ratio of the coating thickness to the substrate thickness

3.5
deflection ratio

ratio of the deflection increment of uncoated substrate to the deflection increment of coated test piece under a given load increment for three-point bending test

4 Symbols

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

Table 1 — Symbols and designations

Symbol	Designation	Unit	References
H	Thickness of substrate	mm	Figure 1 Formula (1)
h	Thickness of coating	mm	Figure 1
B	Width of test piece	mm	Figure 1
P	Peak load	N	Figure 2
L	Span of test piece	mm	Formula (1) Formula (5)
E	Elastic modulus	GPa	Formula (1)
E_c	Elastic modulus of coating	GPa	Formula (2)
E_s	Elastic modulus of substrate	GPa	Formula (2)
α	Ratio of the elastic modulus of the coating to that of the substrate		Formula (2) Formula (5)
f	Deflection	mm	Formula (1)
σ_c	Bending strength of coating	MPa	Formula (5)
P_c	Critical fracture load	N	Formula (5)
y_c	Distance from the tensile surface to the neutral axis	mm	Formula (5)
I	Moment of inertia of the test pieces	mm^4	Formula (5)
ΔP	Load increment	N	Formula (1)
Δf	Deflection increment	mm	Formula (1)
n	Effective test number	numerical	Formula (3) Formula (4) Formula (6) Formula (7)
$\bar{\sigma}$	Mean value of bending strength	MPa	Formula (6) Formula (7)
σ_i	Bending strength of the *i*th test piece	MPa	Formula (6) Formula (7)
$\bar{E}$	Mean value of elastic modulus	GPa	Formula (3) Formula (4)
E_i	Elastic modulus of the *i*th test piece	GPa	Formula (3) Formula (4)
s_e	Standard deviation of measured elastic modulus	GPa	Formula (4)
s_σ	Standard deviation of measured bending strength	MPa	Formula (7)

5 Principle

The elastic modulus and bending strength of thick ceramic coatings on metal or ceramic substrates can be evaluated using three-point bending tests. The elastic modulus of the coating is deduced by comparing the deformation of a coated test piece and of the uncoated substrate under identical loads. A precondition of this method is that the elastic modulus of the substrate is known or can be measured before or after the test. The bending strength of the coating is determined using the critical load for cracking in the coating and the sample size. This indirect test method is called the relative method.

6 Apparatus

6.1 Testing machine

A suitable testing machine capable of applying a uniform crosshead speed and compliant with ISO 7500-1 shall be used. The loading speed should be constant. The measuring error shall be 1 % or lower.

6.2 Data acquisition

Record the applied load as a function of crosshead displacement or testing time in order to determine the maximum applied load.

An analog chart recorder or digital data collection system should be used. The error of the recording system shall be 1 % or lower. The minimum data collection frequency shall be 15 Hz, and a response frequency of 50 Hz is deemed adequate.

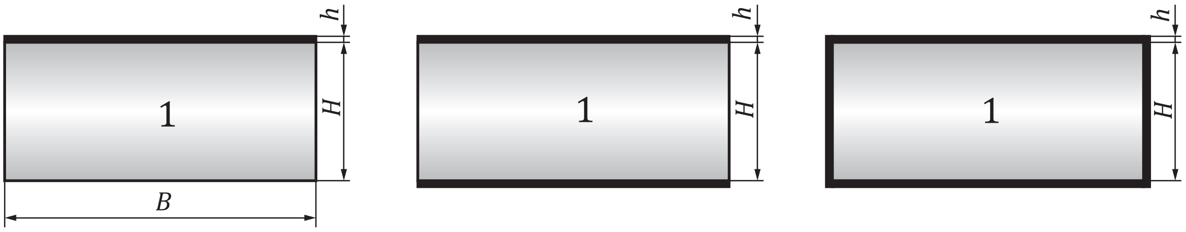
6.3 Dimensional measuring devices

The dimensions of the test piece shall be measured using a Vernier caliper complying with ISO 3611 and with a precision of 0,02 mm or better, or other calibrated measuring device providing the same or better measurement accuracy. Coating thickness shall be measured by using a calibrated optical microscope with magnification of 1 000 times or better. Sample displacement shall be measured using a calibrated electronic micrometer with a precision of at least 0,001 mm and resolution of 0,000 5 mm or better, or other measuring device providing the same or better measurement accuracy. All calibrations shall be traceable to national standards.

7 Test pieces

7.1 Test piece size

In order to simplify the preparation of test pieces, rectangular section test pieces with three different coating configurations are considered: coating on lower surface of the test pieces only [single-face coating, Figure 1 a)], coating on upper and lower surfaces of the test pieces only [two-face coating, Figure 1 b)] and coating on four surfaces of a test pieces [around coating, Figure 1 c)]. Any of the three coating configurations may be used for evaluating the properties of the coating layer. The geometrical dimensions of coated test piece are displayed in Figure 1. Test piece dimensions shall be 36 mm long, 4 mm wide and 2 mm thick or larger with the same dimensional ratio. The thickness ratio, *h*/*H*, should be larger than 1/100. The thickness of the coating shall be larger than 20 µm.



a) Single-face coating **b) Two-face coating** **c) Around coating**

Key

1 substrate

Figure 1 — Schematic of cross-section of test piece with different coating configurations

7.2 Test piece preparation

7.2.1 Test piece machining

The test pieces may be obtained from two approaches.

a) The test pieces are cut from some coated components, carefully grinding and polishing the test piece to keep the surfaces parallel and flat.

b) The test pieces are prepared by coating a substrate; in this case, the modulus of the substrate shall be measured before preparing coating.

The detail test procedure is described below.

Before applying the coating, mark each test piece substrate with a unique identifier which will be visible after coating. Measure the flatness of each uncoated test piece, for example, by mounting in an unstressed state on the x-y stage of a calibrated optical microscope and measuring the z coordinate of the surface with an accuracy of ±2 μm at 10 equally spaced positions along its length. Record the results for each test piece.

For both single-face and two-face coating, carefully mask the faces to remain free from coating, ensuring that the masking material does not prevent the coating from completely covering the faces to be coated. Coat the test pieces using the processing conditions of interest, taking care to obtain the same coating thickness on all faces of interest. If necessary, coat an extra test piece using the same processing parameters and measure the coating thickness on this to determine coating uniformity prior to starting modulus and bending strength measurements. If the observed non-uniformity in coating thickness on different faces is greater than 5 % between the thickest and the thinnest values measured, new test piece should be prepared. Remove the masking from the uncoated faces and repeat the flatness measurement for each test piece and record the results.

7.2.2 Test piece handling and storage

The test pieces shall be handled with care to avoid the introduction of damage after test piece preparation. The 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 6 test pieces are required for the test; the maximum load for fracture could be estimated by using the first test piece.

Minimum of 30 test pieces is recommended if a statistical bending strength analysis (e.g. a Weibull analysis) is to be made. The use of 30 test pieces will help obtain good confidence limits for the elastic modulus and bending strength distribution parameters including a Weibull modulus.

8 Test procedure

8.1 Testing machine and loading speed

Use a universal mechanical testing machine with a crosshead speed of 0,5 mm/min for the three-point bending tests.

8.2 Elastic modulus measurement

The elastic modulus of the substrate should be known or should be measured using an uncoated sample.

The elastic modulus of a homogenous material can be calculated through the ratio of load increment, ΔP, to deflection increment, Δf, at the mid-span of a rectangular beam specimen in three-point bending.

$$E = \frac{L^3}{4H^3 B \cdot 1\,000} \cdot \frac{\Delta P}{\Delta f} \tag{1}$$

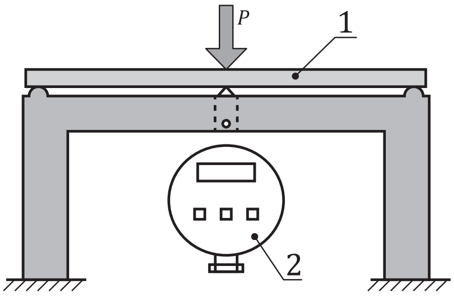
where

E	is the elastic modulus, in GPa;
L	is the span of test pieces, in mm;
H	is the thickness of test pieces, in mm;
B	is the width of test pieces, in mm;
ΔP	is the load increment within the scope of elastic deformation of the substrate, in newtons (N);
Δf	is the equivalent deflection increment, in mm.

Mount the first test piece in the three-point bend apparatus, ensuring that any curvature detected after coating is concave on the side to which the bending load will be applied. Bring the loading head into contact with the test piece mid-way between the two mounting rollers, ensuring uniform contact between the pressure head and the test piece. Bring the probe of the deflection measuring device (electronic micrometer) into contact with the opposite side of the test piece and directly opposite the pressure head, ensuring a positive deflection reading is obtained; see Figure 2. Record this reading.

Apply a test load incrementally from 0,1 P_c to 0,5 P_c (or less, if necessary, to maintain the bending of the test piece in the elastic regime of the substrate) at the specified rate, recording the incremental deflection, with an accuracy of 0,001 mm or better, as a function of incremental load, measured with an accuracy of ±1 % or better.

Calculate the elastic modulus of the coated test piece using Formula (1) and the equivalent incremental load and incremental deflection measurements.



Key

1	specimen
2	micrometer

Figure 2 — Schematic of measurement of load-deflection relation in three-point bending test

8.3 Bending strength measurement

To measure the bending strength of the coating, put each test piece in the fixture, as shown in Figure 2. Ensure uniform contact between the pressure head and the test piece. Apply the test force at the specified rate and record the peak load, P, during the fracture process. The bending strength of the ceramic coating can be calculated from the critical load and sample size. Measure the peak load with an accuracy of ±1 % or better. The acoustic emission technique is recommended to determine the critical load for the test piece with metal substrate. Note that the critical load would be the peak load in many cases of brittle substrate.

8.4 Coating thickness measurement

Following the bending strength measurement on the first test piece, measure the coating thickness on all coated faces to ensure the thickness uniformity is better than 10 % between the thickest and the thinnest values measured. If the uniformity is worse than this, it will be necessary to prepare new test pieces with better coating uniformity. The use of one face coating will help to avoid this issue.

Care needs to be taken with the preparation of the side of the sample so that good contrast is obtained between the coating and substrate so that good measurements of coating thickness can be obtained.

8.5 Temperature and relative humidity

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

9 Calculation of results

9.1 Calculation of elastic modulus

9.1.1 Calculation of elastic modulus in bending test

The elastic modulus of coating, E_c, is given by Formula (2):

$$E_c = E_s \times \alpha \tag{2}$$

where

E_s is the elastic modulus of the substrate;

α is the ratio of the elastic modulus of the coating to that of the substrate.

Under a fixed load increment, the deflection increment for a substrate sample is f_1 before coating and f_2 after coating, which can be measured by electronic micrometer as shown in Figure 2. The modulus ratio, α, for three coating test pieces is shown in Table 2. If the modulus and the dimensions of the substrate is known, the deflection under a given three-point bending load, f_1, can be calculated based on Formula (1), without test.

Table 2 — Relationship between the modulus ratio and deflection ratio for three coating test pieces

No.	Different coating configurations	α value
1	Single-face coating [Figure 1 a)]	$\alpha = \frac{-A+\sqrt{A^2+C}}{2R^3}$ where $R = h/H$; $F = f_1/f_2$; $A = 4R^2+6R+4-F$; $C = 4R^2(F-1)$.
2	Two-face coating [Figure 1 b)]	$\alpha = I_0\left(\frac{f_1}{f_2}-1\right)\bigg/\left[\frac{Bh^3}{6}+\frac{Bh(h+H)^2}{2}\right]$
3	Around coating [see Figure 1 c)]	$\alpha = I_0\left(\frac{f_1}{f_2}-1\right)\bigg/\left[\frac{h(2h+H)^3}{6}+\frac{Bh^3}{6}+\frac{Bh(h+H)^2}{2}\right]$

where

$I_0 = \frac{BH^3}{12}$ is the moment of inertia of the original sample without coating, in mm^4;

f_1 is the deflection increment for a substrate sample without coating, in mm;

f_2 is the deflection increment for a substrate sample after coating, in mm;

h is the thickness of coating, in mm;

H is the thickness of the substrate sample, in mm;

B is the width of the substrate sample, in mm.

The modulus of the coating is determined according to following steps.

a) Measure the deflection increment f_1 and the elastic modulus, E_s.

b) Measure the deflection increment f_2 with the same load increment.

c) Determine the modulus ratio, α, based on Table 2.

d) Calculate the modulus of the coating, E_c, by Formula (2).

If only the coated samples are available, step b) should be carried out first, and then the coating layer should be removed, e.g. by grinding, to expose the substrate, then step a) should be done. It is essential that $f_2 < f_1$ under same ΔP.

9.1.2 Mean value and standard deviation for elastic modulus

The mean elastic modulus, $\overline{E}$, and the standard deviation, s_e, are given by Formula (3) and Formula (4):

$$\bar{E} = \frac{\sum_{i=1}^{n} E_i}{n} \tag{3}$$

$$s_e = \left[\frac{\sum_{i=1}^{n} \left(E_i - \bar{E} \right)^2}{n-1} \right]^{1/2} \tag{4}$$

where

E_i is the elastic modulus of the ith test piece;

n is the total number of test piece.

9.2 Calculation of bending strength

9.2.1 Calculation for bending strength of the ceramic coating

The bending strength of the ceramic coating is calculated using Formula (5):

$$\sigma_c = \frac{\alpha P_c \cdot L}{4I} \times y_c \tag{5}$$

where

α is the ratio of the elastic modulus of coating to that of substrate, determined using the formulae given in Table 2;

P_c is the critical load for crack initiation in the coating, in newtons (N);

L is the span in three-point bending test, in mm;

y_c is the distance from the tensile surface to the neutral axis, in mm, obtained from the formulae in Table 3 for the different coating configurations;

I is the moment of inertia of the test pieces, in mm^4, obtained from the formulae in Table 3 for the different coating configurations.

Table 3 — Summary of the formulae for the distance from the tensile surface to the neutral axis, y_c, and moment of inertia, I, of test pieces for different coating configurations.

No.	Coating configurations	y_c value	Value of I
1	Single-face coating [see Figure 1 a)]	$\frac{H(H+2\alpha h)+\alpha h^2}{2(H+\alpha h)}$	$I=\frac{BH^3}{12}+\frac{\alpha B_1 h^3}{12}+BH\left[\frac{\alpha h(H+h)}{2(\alpha h+H)}\right]^2+\alpha Bh\left[\frac{H^2+Hh}{2(\alpha h+H)}\right]^2$
2	Two-face coating [see Figure 1 b)]	$0{,}5H+h$	$I=\frac{\alpha Bh^3}{6}+\frac{\alpha Bh}{2}(h+H)^2+\frac{BH^3}{12}$
3	Around coating [see Figure 1 c)]	$0{,}5H+h$	$I=\frac{\alpha h(2h+H)^3}{6}+\frac{\alpha Bh^3}{6}+\frac{\alpha Bh}{2}(h+H)^2+\frac{BH^3}{12}$

The elastic modulus of the coating should be determined prior to the strength calculation, since α is used in the calculation. The influence of possible residual stress on the coating layer is not considered here.

9.2.2 Mean value and standard deviation for bending strength

The mean bending strength and the standard deviation are given by Formula (6) and Formula (7):

$$\bar{\sigma}=\frac{\sum_{i=1}^{n}\sigma_i}{n} \tag{6}$$

$$s_\sigma=\left[\frac{\sum_{i=1}^{n}(\sigma_i-\bar{\sigma})^2}{n-1}\right]^{1/2} \tag{7}$$

where

σ_i is the bending strength of the ith test piece;

n is the total number of the test pieces.

10 Analysis of precision and uncertainty

The precision of the modulus and strength measurement of ceramic coatings may be effected by many factors, e.g. the uniformity of the thickness, effects of the thickness ratio and the changes of the test condition. Under a given load increase, the measured deflection of a coated test piece should be smaller than that of the test piece without coating, i.e. $f_2 < f_1$. Otherwise, the resultant modulus would be minus.

If the measured deflection increments indicates $f_2 > f_1$ under a given load increment, a most possible reason is that the substrate of two test pieces are not the same one. The coated test piece and the uncoated test piece should own identical substrate, rather than two substrates with the same sizes, because there is a possibility that the stiffness of the uncoated sample is different from the substrate of the coated piece.

In some cases, there is a transfor layer between the ceramic coating and the substrate. If the transfor layer is generated during the coating process and its properties are not the same as the substrate, it is better to test the coated piece first, then grind off the coating and take the remains as a composite substrate.

11 Test report

The test report shall contain at least the following information:

a) the name and address of the testing establishment;

b) the date of the test, customer name and address, and signatory;

c) a reference to this document, i.e. ISO 19603;

d) the test piece shape, size, and the thickness ratio;

e) a description of the test material (material type of the substrate and the coating);

f) the number of tests carried out and the number of valid results obtained;

g) the valid results, mean value, and standard deviations of the elastic modulus and bending strength;

h) the temperature and humidity of the laboratory.

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