INTERNATIONAL STANDARD

ISO 20343

First edition
2017-01

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for determining elastic modulus of thick ceramic coatings at elevated temperature

Céramiques techniques — Méthode d’essai relative à la détermination du module élastique des revêtements de céramique épais à température élevée

Reference number
ISO 20343:2017(E)

Contents

Page

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on 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 the following URL: www.iso.org/iso/foreword.html.

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

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for determining elastic modulus of thick ceramic coatings at elevated temperature

1 Scope

This document specifies a method of test for determining the elastic modulus of thick ceramic monolayer coatings (thickness > 0,03 mm) at elevated temperatures, using the impulse excitation method. 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 brittle ceramic coatings on ceramic or metal substrates. This test method can be used for material research, quality control, and 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 17561, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for elastic moduli of monolithic ceramics at room temperature by sonic resonance*

IEC 60584-1, *Thermocouples — Part 1: EMF specifications and tolerances*

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 plane stress to plane strain

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

3.2
flexural vibrations
vibrations that occur when the displacements in a slender rod or bar are in a plane normal to the length dimension

3.3
thickness ratio
ratio of the coating thickness to the substrate thickness

4 Principle

The elevated temperature elastic modulus of ceramic coatings on metal or ceramic substrates is deduced by comparing the elastic moduli of coated and uncoated samples, under the precondition that the interface between the coating and the substrate is continuous and without a debonding zone. The elastic modulus of ceramic coatings can be calculated from three parameters, i.e. the elastic modulus of the uncoated sample or substrate (E_s), equivalent elastic modulus of the coated sample (E_q), and the ratio of the coating thickness to the substrate thickness ($R = h/H$). The value of E_q and E_s are determined by the impulse excitation technique.

5 Apparatus

5.1 Elastic modulus testing machine

A suitable impulse excitation testing system capable of applying impulses at elevated temperatures shall be used, which shall be in accordance with ISO 17561. Since the test piece is required by ISO 17561 to be homogeneous and isotropic, the measured modulus using the coated test piece is then described as an equivalent elastic modulus.

5.2 Heating system

5.2.1 General

The heating system, usually a furnace, shall be capable of heating the test fixture and test piece and maintaining a uniform and constant temperature during the impulse excitation test. The furnace shall have the capability for containing air, inert gas, or a vacuum environment as required for the test.

5.2.2 Temperature stability

The furnace shall be controlled by a device capable of maintaining a constant temperature within ±2 °C or better within its working space during the time when the elastic modulus of test pieces is being measured.

5.2.3 Test temperature uniformity

The furnace shall be capable of maintaining a uniform test piece temperature. It shall previously be determined that the temperature of the test piece does not vary by more than 0,5 % of the test temperature after a 15 min hold time at the required test temperature measured in °C.

5.2.4 Furnace heating rate

The furnace control device shall be capable of maintaining a heating rate of the furnace of 5 °C/min to 10 °C/min and preventing temperature overshoots.

5.2.5 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.

5.3 Temperature measuring and indicating instruments

5.3.1 General

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

5.3.2 Thermocouples

Thermocouples in accordance with IEC 60584-1 shall be used. The thermocouple shall exhibit low thermal inertia (the diameter of the wires shall not be greater than 0,5 mm). The measuring thermocouple tip shall be less than 2 mm from the test piece, but never contact the test piece.

5.3.3 Verification of the thermocouple temperature measuring system

Thermocouples shall be calibrated periodically against national standards since calibration may drift with usage or contamination.

5.4 Dimension-measuring device

A Vernier caliper with a precision of 0,02 mm according to ISO 3611 should be used to measure the dimensions of the test piece. The thickness of the coating shall be measured by using a traceably calibrated optical or scanning electron microscope (SEM) with magnification of 1 000 times or better.

5.5 Test fixture

5.5.1 General

The fixture shall be suitable for the installation of the test piece as recommended in ISO 17561 with platinum hanging wires and for impulse excitation and signal acquisition in the furnace. The fixture shall maintain the position of the test piece during the test.

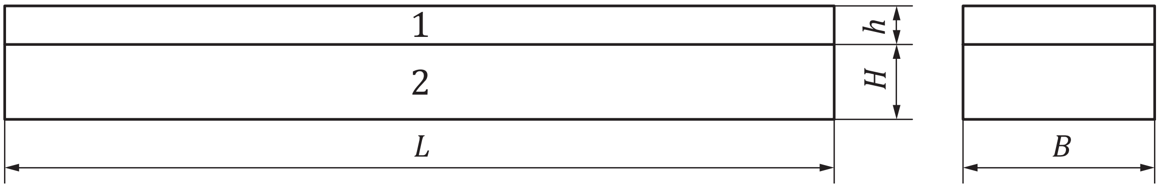
5.5.2 Fixture material

The fixture shall be manufactured from a material that is inert under the testing conditions used. The fixture shall be oxidation-resistant if the testing is conducted in air. The fixture shall have negligible chemical reaction with and shall not contaminate the test piece.

6 Test pieces

6.1 Test piece size

A rectangular test bar with coating on one of the two largest faces only is required. The geometrical dimensions of the coated sample are displayed in Figure 1. The thickness ratio, R ($R = h/H$), shall be larger than 1/100. For flexural resonance, the length of the specimen (L) shall be greater than 40 mm. The ratio of length to thickness of substrate, L/H, shall be greater than 20. The ratio of width to thickness of substrate, B/H, shall be in the range of 3 to 10. The parallelism tolerance on opposite longitudinal faces is 0,015 mm. Table 1 shows the recommended dimensions of a ceramic-coated test piece. The surface of the test piece shall be smooth and flat. The surface shall be ground using a fine grit (400 grit or finer). Any machining procedure used shall not affect the results. The edges of the test piece shall not be chamfered.



Key

1 coating

2 substrate

Figure 1 — Schematic illustration of the rectangular test pieces

Table 1 — Recommended dimensions for coated test piece

Dimensions in millimetres

Dimension	Description	Value	Tolerance
L	Length of the bar	>40	±0,5
B	Width of the bar	8 to 20	±0,1
$H + h$	Total thickness of the bar	2 to 4	±0,05
An approximate ratio for L:B:(H + h) = 20:5:1 is recommended for elastic modulus measurement of thick ceramic coating with h > 0,03 mm and 1 > h/H > 0,01.			

6.2 Test piece preparation and storage

The test pieces may be obtained using one of the 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 depositing the coating. The detailed 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.

The test pieces shall be handled carefully 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.

A minimum of 5 test pieces is required for the test.

7 Test procedure

7.1 Measurement of the size and mass of the test-pieces

Dry the test pieces until their mass is constant. Weigh each of the test pieces to the nearest 1 mg or 0,1 % (whichever is greater) using a calibrate scale. Measure the length of coated test piece to the nearest 0,05 mm or 0,1 % (whichever is greater). Measure the thickness of coating and the substrate to nearest 0,001 mm or 0,1 % (whichever is greater).

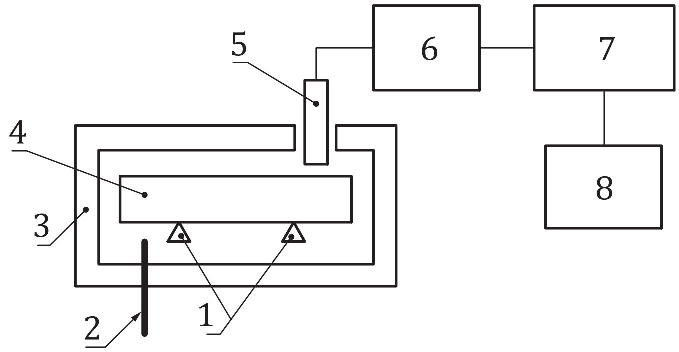
Measure the dimensions of the test pieces using a Vernier caliper complying with ISO 3611 and with precision of 0,02 mm or better, or other calibrated measuring device providing the same or better measurement accuracy. Measure the width and thickness at three points along its length according to 5.5 (at the middle and two ends of the bar) and determine the average of the three measurements. Coating thickness shall be measured by using a calibrated optical or scanning electron microscope (SEM) with magnification of 1 000 times or better.

The variation in coating thickness between the thickest and thinnest values measured shall be less than or equal to 10 %. If the variation is more than this, prepare new test pieces meeting this requirement.

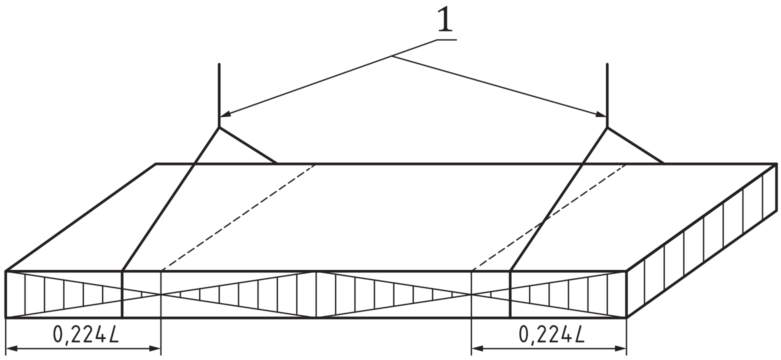
Care needs to be taken with the preparation of the side of the sample so that good contrast is obtained between the coating and substrate so that good measurements of coating thickness can be obtained.

7.2 Measurement of elastic modulus of coating at elevated temperature

Suspend one of the test pieces, supported by a heat-resisting material like platinum wire in accordance with ISO 17561, as shown in Figure 2, in the furnace. The suspending devices shall permit the free vibration of the test piece. The elastic modulus of the substrate shall be known, or be measured at the temperature or temperatures of interest, using an uncoated sample.



a) Impulse excitation testing method



b) Recommended support system

Key

1 support system
2 impulser
3 furnace
4 specimen
5 transducer
6 signal amplifier
7 frequency analyzer
8 results output

Figure 2 — Schematic illustration of impulse excitation testing method and recommended support system

Heat the test piece to the testing temperature and hold it at this temperature for a sufficient time (15 min is recommended) for the specimen temperature to reach equilibrium. The time allowed for this shall be stated in the test report. The temperature measured by the thermocouple or other measuring device shall not vary during the time of the testing by more than ±0,5 % of the test temperature measured in °C. Excite flexural resonance of the samples by applying an impulse and measure the frequency of the resonance generated. Determine the elastic modulus of substrate, E_s, and the elastic modulus of coated test pieces, E_q, according to ISO 17561, respectively. Test results shall be obtained from at least 5 coated test pieces, all of which were coated at the same time by the same process.

8 Calculation of results

8.1 Standard formula for elastic modulus of coating

The standard formula for the elastic modulus of the coating, E_c, is given by Formula (1):

$$E_c = \frac{-A + \sqrt{A^2 + C}}{2R^3} \cdot E_s \quad (1)$$

where

$$R = h / H \quad (2)$$

$$F = (1 + R)^3 \cdot (E_q / E_s) \quad (3)$$

$$A = 4R^2 + 6R + 4 - F \quad (4)$$

$$C = 4R^2 \cdot (F - 1) \quad (5)$$

where

h is the thickness of ceramic coating, in mm;

H is the thickness of the substrate, in mm;

E_q is the elastic modulus of coated specimen, in GPa;

E_s is the elastic modulus of substrate, in GPa.

Determine the thickness ratio, R, using Formula (2) and the stiffness ratio of the substrate sample to the coated sample, F, using Formula (3). Calculate the values of A and C using Formula (4) and Formula (5), respectively. The elastic modulus of the coating, E_c, can then be obtained using Formula (1).

Thus, the test procedure for coating's modulus has three steps.

a) Measure the modulus of coated test piece varying with temperature.

b) Test the modulus of the substrate varying with temperature.

c) Calculate the modulus of the coating at different temperatures.

8.2 Mean value and standard deviation for elastic modulus

The mean elastic modulus, $\overline{E}$, and the standard deviation, s, are given by Formula (6) and Formula (7):

$$\bar{E} = \frac{\sum_{i=1}^{n} E_i}{n} \tag{6}$$

$$s = \left[\frac{\sum_{i=1}^{n} \left(E_i - \bar{E} \right)^2}{n-1} \right]^{1/2} \tag{7}$$

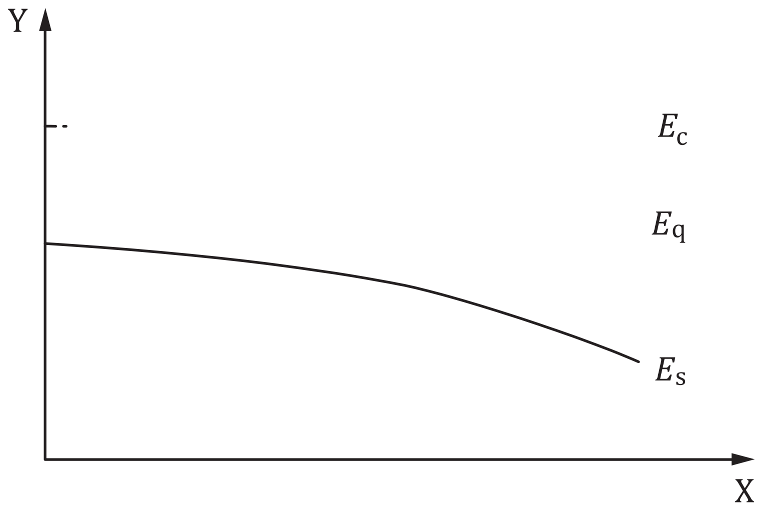
where

E_i is the elastic modulus of the ith test piece;

n is the total number of test pieces.

8.3 Variation of elastic modulus with temperature

As it is unlikely that the temperature of operation of the ceramic coating will be well-established or even constant, it is usual to determine the elastic moduli of coated and uncoated specimens under a range of temperatures to enable a plot of modulus versus temperature to be obtained. At each testing temperature, the elastic modulus of the ceramic coating should be deduced, from which the curve of the relationship between the elastic modulus of the coating and testing temperatures can be derived. Figure 3 shows schematic results of elastic modulus for coated sample (E_q) and substrate (E_s) at different temperatures by the impulse excitation tests and the calculated elastic modulus of the coating (E_c) at these temperatures.



Key

X testing temperature (°C)

Y elastic modulus (GPa)

Figure 3 — Schematic results of elastic modulus of coating (E_c) changed with the elastic modulus of coated test piece (E_q) and substrate (E_s) at different temperatures (T)

9 Analysis of precision and uncertainty

The precision of the modulus measurement of the coating involves many factors, e.g. the uniformity of the thickness, effects of the thickness ratio, and interface defects. In order to get the reasonability of the calculation, it is supposed that the interface bonding between the coating and substrate is continuous and uniform. However, in many cases, the interface bonding is not perfectly continuous, which results in a little lower data of the measured modulus. This uncertainty problem for the impulse excitation tests is more sensitive than the three-point bending tests.

The precision of measurement of fundamental mode natural frequency, dimensions, and mass of the test pieces should meet the requirements as described in ISO 17561.

10 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 20343;

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

e) a description of the substrate material (material type and any pre- and post-treatment of the substrate) and of the coating and coating process;

f) the test temperature, type of heating element, temperature measuring device;

g) the furnace environment: air, vacuum, or inert atmosphere;

h) the approximate rate of heating and hold time at the test temperature prior to the test;

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

j) the valid results, mean value, and standard deviations of the elastic modulus;

k) the temperature and humidity of the laboratory.

Bibliography

[1] ISO 18558, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for determining elastic modulus and bending strength of ceramic tube and rings*

[2] Bao Y.W., Zhou Y.C., Bu X.X. Evaluating elastic modulus and strength of hard coatings by relative method. *Mater. Sci. Eng. A*. 2007, **458** (1-2) pp. 268–274

[3] Bao Y.W., Wang W., Zhou Y.C. Investigation of the relationship between elastic modulus and hardness based on depth-sensing indentation measurements. *Acta Mater.* 2004, **52**, pp. 5397–5404

[4] Bao Y.W., & Zhou Y.C. Evaluating high-temperature modulus and elastic recovery of Ti_3SiC_2 and Ti_3AlC_2 ceramics. *Mater. Lett.* 2003, **57**, pp. 4018–4022

ICS 81.060.30

Price based on 9 pages