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

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 20343 was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

This second/third/... edition cancels and replaces the first/second/... edition (), [clause(s) / subclause(s) / table(s) / figure(s) / annex(es)] of which [has / have] been technically revised.

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

1 Scope

This standard 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 International Standard 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, 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 17561, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for elastic moduli of monolithic ceramics at room temperature by sonic resonance*

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

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
elastic modulus
ratio of plane stress to plane strain, 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, viz. the elastic modulus of the uncoated sample or substrate (Es), equivalent elastic modulus of the coated sample (Eq), and the ratio of the coating thickness to the substrate thickness (R=h/H). The value of Eq and Es 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 ℃ 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 minutes 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 5-10 ℃/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

Thermocouple temperature measuring equipment shall have a resolution of at least 1 ℃ and an accuracy of 5 ℃ or better. Optical pyrometers, if used, shall have a resolution of at least 5 ℃ and an accuracy of 5 ℃ 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 electron microscope with magnification of 1000 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.

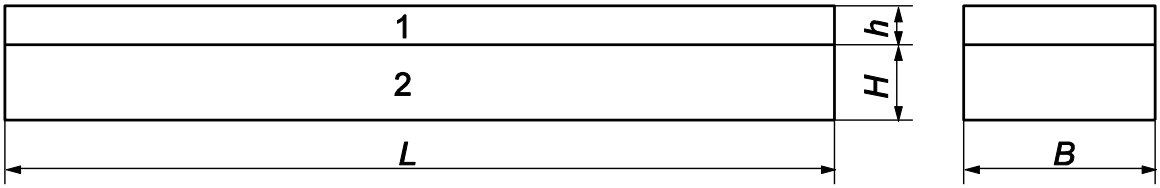
5.5.2 Fixture material

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 2 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 2– Recommended dimensions for coated test piece

In millimeter

Dimension	Description	Value	Tolerance
L	Length of the bar	›40	±0,5
B	Width of the bar	8-20	±0,1
H+h	Total thickness of the bar	2-4	±0,05

Note 1 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 two approaches: 1) The test pieces are cut from some coated components, carefully grinding and polishing the test piece to keep the surfaces parallel and flat

2) 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. Test procedure is described as 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, e.g. 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. 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 microscope with magnification of 1000 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.

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.