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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for determining density of ceramic coatings

Céramiques techniques (céramiques avancées, céramiques techniques avancées) — Méthode d'essai pour déterminer la densité des revêtements céramiques

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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 density of ceramic coatings

1 Scope

This document specifies a testing method for determining the density of thick ceramic coatings at ambient temperature using the Archimedes method. Methods for test piece preparation, test modes, data collection and density calculation are addressed.

This document applies to ceramic coatings with a thickness > 0,1 mm including thermal barrier coatings, wear-resistance coatings and other thick coatings on metal or ceramic substrates.

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 18754, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of density and apparent porosity*

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
volume
apparent solid volume, the sum of the respective volumes of the solid material

3.2
density
apparent solid density, the ratio of the mass of the dry material to its apparent solid volume

4 Principle

The density of ceramic coating on a substrate is determined by comparing the densities of the coated and uncoated sample, under the precondition that the interface between the coating and the substrate is continuous and without a debonding zone. The density of the ceramic coating is obtained from three parameters: i) the density of the uncoated substrate (ρ_S); ii) the equivalent density of the coated sample ($\bar{\rho}$); and iii) the ratio of the dry weight of the substrate to that of the coated sample ($k = m_S/m$). The values of ρ and $\bar{\rho}$ are determined by the Archimedes method according to ISO 18754, and they are related to the density of the coating via a unique formula.

5 Apparatus

5.1 Density testing apparatus

The apparatus for density tests shall conform to the requirements of ISO 18754.

5.2 Dimension measuring device

A vernier calliper with a precision of 0,02 mm according to ISO 3611 shall be used to measure the overall dimensions of the sample. The thickness of the coating and the substrate shall be measured by using an optical or electronic microscope with a magnification of 1 000 times or more.

6 Test pieces

6.1 Type of test piece

Test pieces are defined as two types according to test requirements and sample conditions.

Type A: rectangular parallelepiped test piece with a single face coating. The density test procedure shall be i) density tests for the coated test piece; ii) grind off the coating; iii) the density test of the remaining body (substrate or composite substrate).

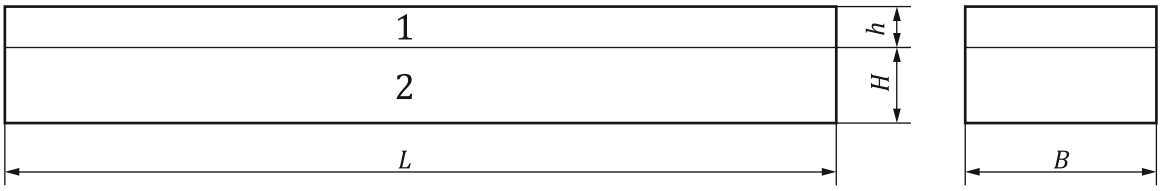
Type B: test piece with various shapes; it is impossible to grind off the coating. The test procedure shall be i) density tests for the substrate; ii) make coating on the substrate; iii) density tests for the coated piece.

The volume of each test piece shall conform to the requirements of ISO 18754. The volume of each individual test piece shall be not less than 0,36 cm^3.

6.2 Test piece preparation

6.2.1 Type A test piece preparation

A rectangular parallelepiped test piece with single-face coating is required. For test pieces with multilayer coatings, the first layer of coatings (top layer) is considered as a single-layer coating and the rest is considered as the substrate. The geometrical dimensions of a coated sample are displayed in Figure 1. Table 1 shows the recommended dimensions of a type A test piece. The surfaces shall be ground using a fine grit (400 grit or finer).



Key

1 coating

2 substrate

Figure 1 — Schematic illustration of a rectangular parallelepiped test piece with ceramic coating

Table 1 — Recommended dimensions for type A test piece in millimetres

Dimension	Description	Value (mm)	Tolerance (mm)
L	Length of the piece	> 30	±0,5
B	Width of the piece	6–15	±0,1
$H + h$	Total thickness of the piece	2–4	±0,05

Any dust and chips liable during further handling shall be removed from the surface of each test piece.

Test pieces shall be smooth and flat, so their surfaces can be dried by wiping to remove any remaining immersion liquid, since roughness limits the accuracy of the measurement of the mass of the soaked test piece.

NOTE For test pieces with multilayer coatings, the density of each layer can be determined by repeating the tests for type A in 7.1.

6.2.2 Type B test piece preparation

The type B test piece has a random and complex shape. It is easy to add a coating but difficult to remove it. Therefore, the density of the substrate shall be measured first, and then the substrate shall be coated according to the required process. Finally, the density of the coated component shall be measured. Thus, the density of the coating is determined via the results of the above two tests.

The volume of each type B test piece shall conform to the requirements of ISO 18754. When the volume of each individual test piece is less than the value specified in ISO 18754, a sufficient number of test pieces shall be taken so that the total volume of the test pieces reaches the minimum volume specified.

6.3 Test piece storage

The test pieces shall be handled carefully to avoid damage after test piece preparation. Test pieces shall be stored separately and not allowed to touch or scratch each other.

6.4 Number of test pieces

A minimum of three test pieces are required for the tests.

6.5 Temperature and relative humidity

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

7 Test procedure

7.1 Type A test piece

7.1.1 Determination of mass and density of coated test piece

Measure the mass of the dry coated test piece and the density of the coated test piece in accordance with ISO 18754.

7.1.2 Determination of mass and density of substrate

Grind off the coating from the type A test piece to obtain the equivalent substrate piece. Then measure the mass of the dry substrate piece and the density of the substrate piece in accordance with ISO 18754.

7.2 Type B test piece

7.2.1 Determination of mass and density of substrate

Measure the mass of the dry substrate piece and the density of the substrate piece in accordance with ISO 18754.

7.2.2 Making coating

After the tests detailed in 7.2.1, add the coating to the substrate according to the requirements of the product.

7.2.3 Determination of mass and density of the coated sample

Measure the mass of the dry coated test piece and the density of the coated test piece in accordance with ISO 18754.

8 Calculation of results

8.1 Calculation of the density of coating

Calculate the density of the coating using the standard Formula (1).

$$\frac{1}{\rho_c} = \left(\frac{1}{\rho_{comp}} - \frac{k}{\rho_s} \right) \frac{1}{1-k} \quad (1)$$

where

k is m_s/m ($k < 1$);

ρ_c is the density of the coating, in g/cm^3;

m_s is the mass of dry substrate test piece, in g;

m is the mass of dry coated test piece, in g;

ρ_{comp} is the density of coated test piece, in g/cm^3;

ρ_s is the density of substrate, in g/cm^3.

8.2 Mean values of coating, substrate and coated test piece density

Calculate the average densities of the coating, substrate and coated test piece, respectively, using Formula (2).

$$\bar{\rho} = \frac{\sum_{i-1}^{n} \rho_i}{n} \quad (2)$$

Where $\bar{\rho}$ is the mean density of the coating, substrate or coated test piece, respectively, ρ_i is the density of the i^{th} test piece, and n is the total number of test pieces.

9 Analysis of precision and uncertainty

The precision of the density measurement of the coating involves many factors, such as the roughness of the surface, the thickness of the coating and the balance precision. The shape of the test piece has no effect on the test precision, but the coating thickness shall not be too thin. If the value of $k = m_s/m$

is equal to or higher than 1, the results are incorrect and the test shall be repeated. The precision of measurement of volume and mass of the test pieces should meet the requirements of ISO 18754.

10 Test report

The test report shall contain at least the following information:

a) a reference to this document;

b) the name and address of the testing laboratory;

c) the mean value of the measured density of the coating, substrate and the coated test piece;

d) a description of the type A or type B test piece, dimension and shape, surface finish of the test piece;

e) the date of the test, the customer name, address and signature;

f) the ambient temperature and relative humidity during the test;

g) the description and test material (material type, manufacturing code, batch number);

h) the number of tests carried out and the number of valid results obtained.