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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for determining thermal expansion coefficient and residual stress of CVD ceramic coatings

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Foreword

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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 thermal expansion coefficient and residual stress of CVD ceramic coatings

1 Scope

This document specifies a test method for determining thermal expansion coefficient up to 2 300 K and the residual stress of chemical vapour deposition (CVD) ceramic coatings (thickness > 0,03 mm) at room temperature. Procedures for test piece preparation, test modes, heat rate, data collection, property calculations and reporting procedures are given.

This document applies to CVD ceramic 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 17139, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Thermophysical properties of ceramic composites — Determination of thermal expansion*

ISO 19603, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for determining elastic modulus and bending strength of thick ceramic coatings*

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:

— ISO Online browsing platform: available at https://www.iso.org/obp

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

3.1
relative method
indirect test method to obtain the measured parameter

Note 1 to entry: The expected parameter A is usually difficult to measure directly, but parameters B and C are known parameters or easy to obtain. If the analytical relationship of A = f (B, C) is established, the parameter A can be calculated.

3.2
linear thermal expansion
positive or negative change in one dimension that occurs when a material is subjected to a change in temperature

4 Principle

The deformation of a coated bar during heating or cooling involves two effect factors, i.e. thermal expansion coefficient and residual stress in the coating and the substrate. The thermal expansion coefficient is a material characteristic. However, the residual stresses in the coating are not only due to a material property but depend also on the size of the component and the cross-sectional area ratio of the coating and substrate.

The thermal expansion coefficient of CVD ceramic coatings is deduced by the relative method, i.e. it is related with the expansion coefficient of the substrate and the coefficient of the coated bar. Two types of test piece, a bar with CVD ceramic coating (composite) and a bar without coating (substrate), shall be used to measure the thermal expansion coefficient according to ISO 17139. After determining the elastic modulus of the substrate and coating, the thermal expansion coefficient of both the composite and the substrate are obtained. The thermal expansion coefficient of CVD ceramic coating shall then be calculated.

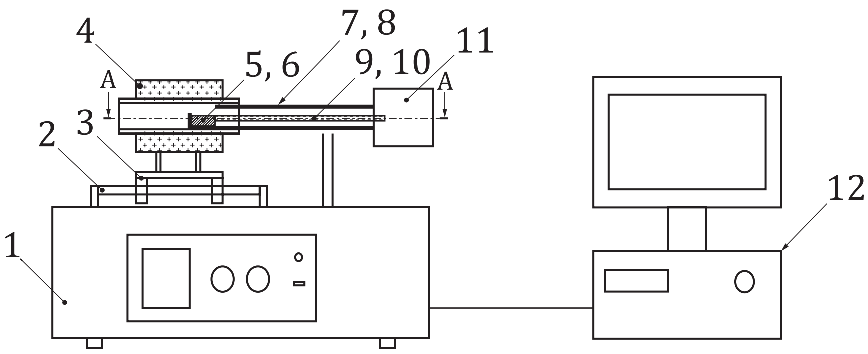
The sample length change and environment temperature are measured continuously at regular, frequent intervals during the imposed cycle. The residual stress of CVD ceramic coatings is usually generated due to the mismatch of thermal expansion coefficient between the coating and the substrate. Based on the uniform strain model, geometric compatibility analysis and internal stress equilibrium, the residual stress calculation formula of CVD ceramic coatings is deduced by the relative method, which is a function of many factors including elastic modulus, expansion coefficient, configurations and temperature. After getting the thermal expansion coefficient of the coating, the residual stress of CVD ceramic coatings shall be calculated from elastic modulus, expansion coefficient, cross-sectional area ratio of the coating to substrate and the temperature during coating preparation.

5 Apparatus

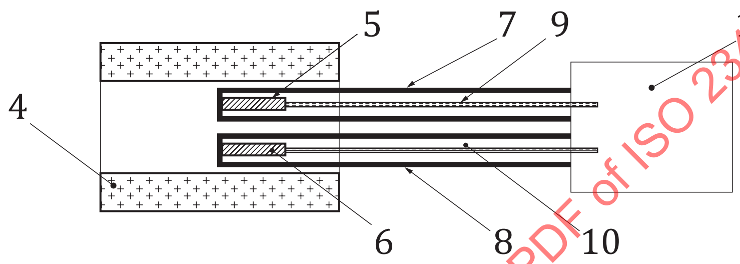
5.1 General

Recommended testing equipment is shown in Figure 1. This can be used to measure the thermal expansion coefficient of two testing pieces in the same environment. If not, the two testing pieces shall be tested respectively in the same environment using the normal thermal expansion equipment in accordance with ISO 17139.

NOTE The two test pieces can be of the same materials or different materials. In this document, one is the composite with CVD coating and the other is the substrate.



a) Front view



b) A - A

Key

1 furnace power control
2 mounting fixed device
3 movable supporting device
4 furnace
5 sample 1
6 sample 2
7 sample 1 holder
8 sample 2 holder
9 push rod
10 high temperature area
11 linear variable differential transformer
12 recording and calculation system

Figure 1 — Schematic of testing equipment

5.2 Heating and cooling device

Furnace, capable of working in a controlled atmosphere when required and of controlling the temperature of the test piece to within 1 % of its mean temperature, expressed in K, in accordance with ISO 17139.

5.3 Temperature measurement

Thermocouple, in accordance with IEC 60584-1, subject to the conditions of the testing environment. For temperatures in excess of 2 000 K, infrared detectors or any suitable device may be used.

5.4 Test piece mounting

The device used shall allow free axial movement of the test piece. The mechanical environment shall minimize stresses. For measuring apparatus that is horizontal, the sideways movement or twist of the test piece shall be restricted, without any restriction of axial movement by a suitable arrangement.

5.5 Data recording system

A system capable of measuring displacements to an accuracy better than 0,000 1 mm. The system shall allow recording of the test piece temperature and the displacement simultaneously.

An analogue 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.

5.6 Dimensional measuring devices

The dimensions of the test piece shall be measured using a Vernier calliper conforming with ISO 3611 and with 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.

6 Specimens

6.1 Test piece

In order to simplify the preparation of test pieces, three different symmetrical structures of coating configurations are considered:

a) coating on upper and lower surfaces of the test pieces only (two-face coating, Figure 2 a);

b) coating on four surfaces of the test pieces (four-face coating, Figure 2 b);

c) coating on cylinder surface of the test pieces (around coating, Figure 2 c). Any of the three coating configurations may be used for evaluating the properties of the coating layer.

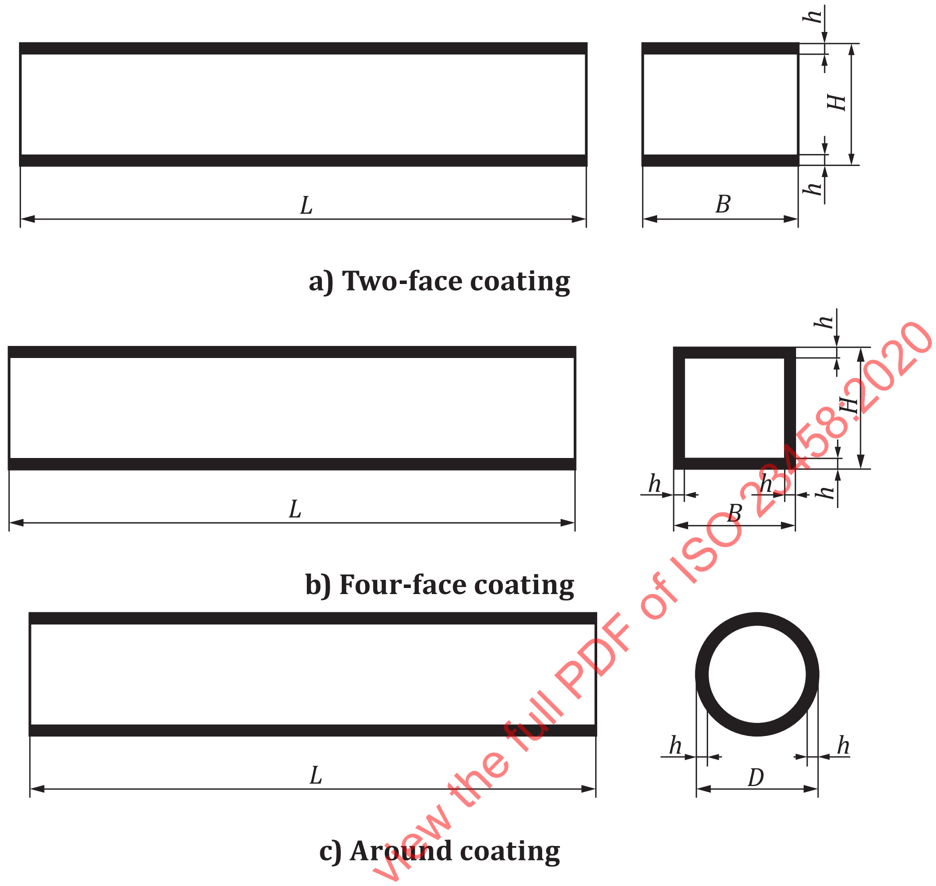


Figure 2 — Schematic of symmetrical structure of test pieces with different coating configurations for the thermal expansion coefficient tests

6.2 Dimensions

The geometrical dimensions of the coated test piece for the thermal expansion coefficient tests are displayed in Figure 2. Table 1 gives the recommended test piece dimensions. The total data are the sum of the coating and substrate for the composite. For coating on a rectangular test piece shown in Figure 2 a) or b), the recommended dimensions shall be 30 mm to 50 mm long, 5 mm wide and 5 mm thick. The thickness of the coating, h, shall be larger than 0,03 mm. The thickness ratio, h/H, should be lower than 1/4. The parallelism tolerance on the opposite longitudinal face shall be 0,015 mm.

For coating on cylinder test pieces shown in Figure 2 c), the recommended dimensions shall be 30 mm to 50 mm long and 5 mm in diameter. The thickness of the coating shall be larger than 0,03 mm. The thickness ratio, h/D, should be lower than 1/4.

It is recommended that a cylinder testing piece is used for testing the thermal expansion coefficient.

Table 1 — Recommended test piece dimensions for the thermal expansion coefficient tests

Dimensions in millimetres

Type	Dimension	Description	Value	Tolerance
Rectangular bar testing piece	L	Total length	30 to 50	±0,5
	B	Total width	5	±0,2
	H	Total thickness	5	±0,2
	h	Thickness of the coating	> 0,03	—

Table 1 *(continued)*

Type	Dimension	Description	Value	Tolerance
Cylinder testing piece	L	Total length	30 to 50	±0,5
	D	Total diameter	5	±0,2
	h	Thickness of the coating	> 0,03	—

6.3 Test piece preparation

6.3.1 General

The test piece preparation is only for the measurement of the expansion coefficient of the CVD coating. Two samples are required for comparison, one bar with uniform CVD coatings and another without coating, conforming with ISO 17139. The non-uniformity of the coating thickness for the test piece shall be lower than 10 %.

Since residual stress is not a material constant, no test piece is required for it.

6.3.2 Test piece handling and 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.

6.3.3 Number of test pieces

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

7 Test procedure

7.1 Measurement of the test piece dimensions

Measure the dimensions of the test pieces using a Vernier caliper conforming with ISO 3611 and with a precision of 0,02 mm or better, or another 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.

The variation in coating thickness between the thickest and thinnest measured values, $(h_{max} - h_{min})/h_{min}$, 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 the elastic modulus

At room temperature, the elastic moduli of the substrate (E_s) and CVD coating (E_c) shall be known or measured in accordance with ISO 19603.

7.3 Measurement of the thermal expansion coefficient of CVD coatings

Measure the thermal expansion coefficient properties of the composite ($\bar{\alpha}$). The substrate (a_s) shall be measured in accordance with ISO 17139. The thermal expansion coefficient of CVD coatings shall be calculated.

7.4 Calculation of the residual stress in CVD coatings

Residual stress in coating is not a material constant and varies with the cross-sectional area ratio of the substrate to coating. For a coated component, the residual stress can be calculated from elastic modulus, expansion coefficient, cross-sectional area ratio of the coating and substrate, and preparation temperature. Both the residual stress of the test pieces and the residual stress of any component with the same coating but different area ratios can be calculated.

For the real structural CVD coatings, the cross-sectional area ratio of the substrate to coating is obtained by using an optical microscope and then the residual stress in coatings is calculated.

8 Calculation

8.1 Thermal expansion coefficient of the CVD ceramic coatings, α_c

Calculate the thermal expansion coefficient of the CVD ceramic coatings in accordance with Formula (1).

$$\alpha_c = \bar{\alpha} - \frac{E_s S_s}{E_c S_c}(\alpha_s - \bar{\alpha}) \quad (1)$$

where

E_s is the elastic modulus of the substrate, in GPa;

E_c is the elastic modulus of the coating, in GPa;

S_s is the cross-sectional area of the substrate, in mm^2;

S_c is the cross-sectional area of the coating, in mm^2;

α_s is the thermal expansion coefficient of the substrate, in K^{-1};

$\bar{\alpha}$ is the thermal expansion coefficient of the composite, in K^{-1}.

8.2 Residual stress in CVD coatings, σ_c

Calculate the residual stress in plane of the CVD ceramic coatings in accordance with Formula (2).

$$\sigma_c = \left(\frac{S_s}{S_c}\right)\left\{1 - \left[\frac{E_s S_s}{E_c S_c} + \frac{\alpha_c}{\alpha_s}\right] \Big/ \left[1 + \frac{E_s S_s}{E_c S_c}\right]\right\} E_s \cdot \alpha_s \cdot \Delta T \quad (2)$$

where

E_s is the elastic modulus of the substrate, in GPa;

E_c is the elastic modulus of the coating, in GPa;

S_s is the cross-sectional area of the substrate, in mm^2;

S_c is the cross-sectional area of the coating, in mm^2;

α_s is the expansion coefficient of the substrate, in K^{-1};

α_c is the thermal expansion coefficient of the coating, in K^{-1};

ΔT is the difference between the preparation temperature of the coating and the room temperature, in K.