INTERNATIONAL STANDARD

ISO 23486

First edition
2021-04

Metallic and other inorganic coatings — Measurement of Young's modulus of thermal barrier coatings at elevated temperature by flexural resonance method

Reference number
ISO 23486:2021(E)

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Published in Switzerland

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Foreword

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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 107, *Metallic and other inorganic coatings*.

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.

Introduction

Thermal barrier coatings (TBCs) are highly advanced material systems, generally applied to surfaces of hot-section components made of nickel or cobalt-based superalloys, such as combustors, blades and vanes of power-generation gas turbines in thermal power plants and aero-engines operated at elevated temperatures.

The function of these coatings is to protect metallic components for extended periods at elevated temperatures by employing thermally insulating materials which can sustain an appreciable temperature difference between load-bearing alloys and coating surfaces. These coatings permit the high-temperature operation by shielding these components, thereby extending their lives.

Although Young's modulus is an important property of TBCs, the existing ISO 19477 only describes a method for measuring this parameter at room temperature.

This document specifies a method for measuring the Young's modulus of TBCs that consist of multilayers formed on substrate by thermal spraying, from room temperature up to elevated temperature.

Metallic and other inorganic coatings — Measurement of Young's modulus of thermal barrier coatings at elevated temperature by flexural resonance method

1 Scope

This document specifies a method for measuring the in-plane Young's modulus of thermal barrier coatings (TBCs) formed on substrates, from room temperature up to 1 000 °C.

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 1463, *Metallic and oxide coatings — Measurement of coating thickness — Microscopical method*

ISO 3611, *Geometrical product specifications (GPS) — Dimensional measuring equipment: Micrometers for external measurements — Design and metrological characteristics*

ISO 13385 (all parts), *Geometrical product specifications (GPS) — Dimensional measuring equipment*

ISO 14188:2012, *Metallic and other inorganic coatings — Test methods for measuring thermal cycle resistance and thermal shock resistance for thermal barrier coatings*

ISO 17561:2016, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for elastic moduli of monolithic ceramics at room temperature by sonic resonance*

ISO 19477, *Metallic and other inorganic coatings — Measurement of Young's modulus of thermal barrier coatings by beam bending*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 14188 and ISO 19477 and the following apply.

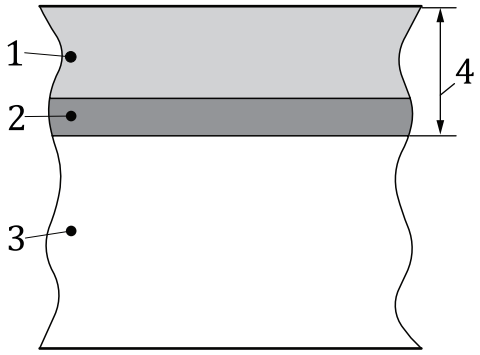
ISO and IEC maintain terminological databases for use in standardization at the following addresses:

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

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

3.1
thermal barrier coating
TBC
two-layer coating consisting of a metallic bond coat (BC) and a ceramic top coat (TC), in order to reduce heat transfer from outside the top coat through the coating to the substrate of a heat-resistant metallic material

Note 1 to entry: See Figure 1.



Key

1 TC
2 BC
3 substrate
4 TBC

Figure 1 — Diagrammatic view of a section of a TBC

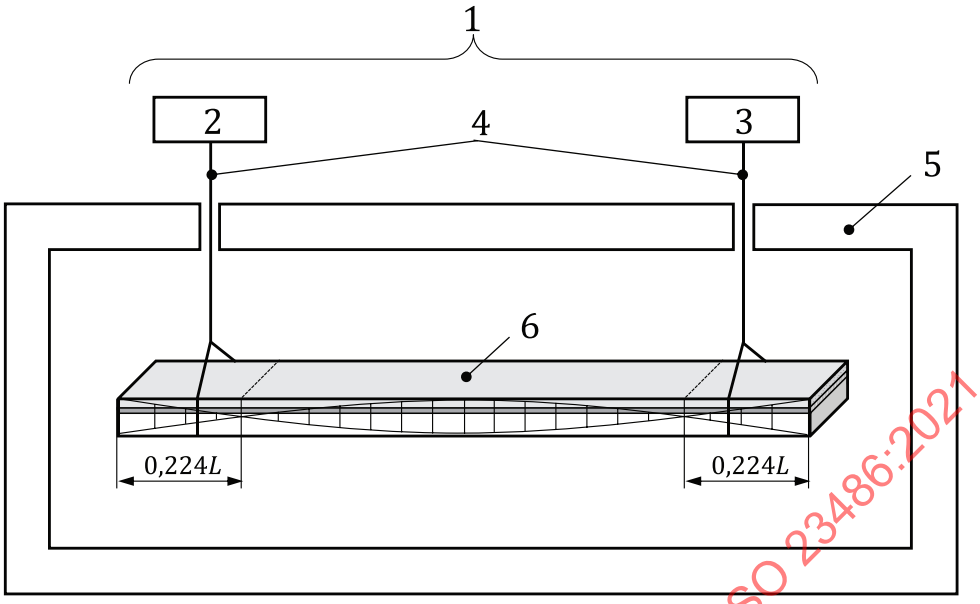
[SOURCE: ISO 14188:2012, 3.1, modified — In the definition, "a ceramic top coat" has replaced "an oxide top coat" and "to the substrate of a heat-resistant metallic material" has replaced "to the base material". A different note to entry and figure have been used.]

4 Principle

The fundamental procedures for measuring the Young's moduli of the substrate, BC and TC consist of the measurement of the resonance frequency of three types of specimens (substrate, substrate with BC and substrate with TBC) at an elevated temperature by a flexural resonance method, and of calculations according to the theory of flexural vibration for a composite beam[1][2][3][4].

5 Apparatus for measuring resonance frequency

An example of the apparatus for measuring the resonance frequency is schematically shown in Figure 2. The apparatus consists of a flexural resonance system and a heating device. See Annex A.



Key

1 flexural resonance system
2 driver
3 detector
4 suspending thread
5 heating device
6 specimen

Figure 2 — Typical apparatus for measuring the resonance frequency

5.1 Flexural resonance system, which shall be specified in accordance with ISO 17561.

The suspending thread shall be a heat-resistant material, such as an alumina yarn.

5.2 Heating device, consisting of a furnace with a temperature control system.

6 Specimen

6.1 Shape and dimensions

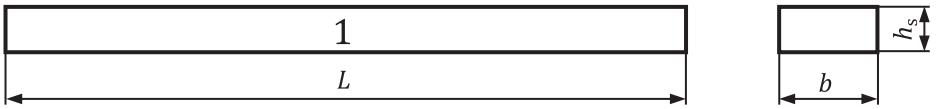
The shape and dimensions of the specimen shall be specified as follows.

a) Three types of specimens (the substrate, BC and TBC) shall be used. The substrate shall be the same as the heat-resistant metallic material to which the TBC is applied.

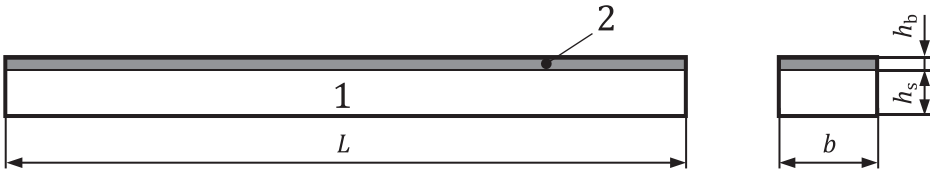
b) The specimen shape is a beam type (see Figure 3) and the dimensions of the specimen shall be as given in Table 1.

c) The thickness tolerance of the substrate shall be ±0,01 mm.

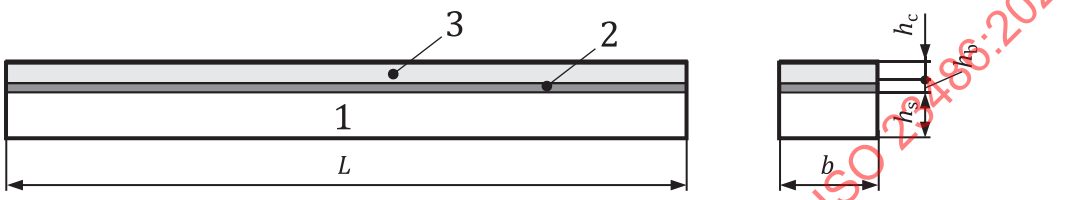
d) The side surface of the BC and TBC specimens shall be polished to remove the coating deposited on the side surface. The polishing shall be done cautiously so that it does not damage the coating.



a) Substrate specimen



b) BC specimen (substrate with BC)



c) TBC specimen (substrate with both BC and TC)

Key

1	substrate	L	total length
2	BC	b	width
3	TC	h_s	substrate thickness
		h_b	BC thickness
		h_c	TC thickness

Figure 3 — Shape of specimens

Table 1 — Dimensions of specimens

Symbol	Designation	Dimension
L (mm)	total length	$70 \leq L$ and $20 \leq L/(h_s + h_b + h_c)$
b (mm)	width	$5{,}0 \leq b \leq (1/6)\ L$
h_s (mm)	substrate thickness	$1{,}5 \leq h_s \leq 2{,}5$
h_b (mm)	BC thickness	$0{,}20 \leq h_b$ and $0{,}10 \leq h_b/h_s$ for BC specimen
h_c (mm)	TC thickness	$0{,}40 \leq h_c$ and $0{,}20 \leq h_c/(h_s + h_b)$ for TBC specimen

6.2 Preheat treatment

Specimens should be heat-treated at the maximum test temperature for about 2 h in air prior to the measurement in order to minimize the change of coating microstructures during the test, since the Young's moduli of as-sprayed BC and TC change remarkably depending on temperature history.

7 Measuring procedure

7.1 Dimension and mass of specimen

The dimensions and the mass of the specimen shall be measured as follows:

a) The total length of the specimen shall be measured in accordance with the ISO 13385 series.

b) The width of the specimen shall be measured in accordance with ISO 3611.

c) The thickness of the substrate shall be measured in accordance with ISO 3611.

d) The thickness of BC and TC shall be measured on an image of the coating cross-section in accordance with ISO 1463.

e) The mass of the substrate, BC and TBC specimens shall be measured.

7.2 Resonance frequency

The resonance frequency of the specimen shall be measured as follows:

a) The specimen shall be suspended by threads connected to the driver and the detector. The suspending method shall permit the free vibration of the specimen.

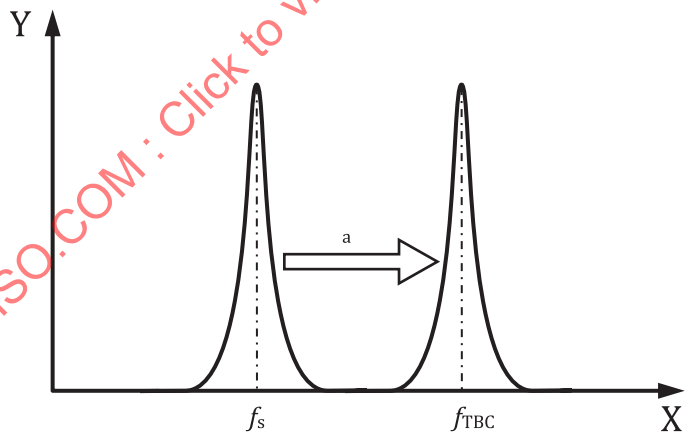
b) The threads shall be positioned outside the nodal points as shown in Figure 2, since the vibration nodes appear at a distance of 0,224 of the total length from each end.

c) The resonance frequency of the substrate specimen shall be measured at room temperature in accordance with ISO 17561.

d) The substrate specimen shall be heated to a specified temperature in air and held for at least 15 min to stabilize the temperature of the specimen.

e) The resonance frequency of the substrate specimen shall be measured at the specified temperature.

f) The resonance frequencies of the BC and the TBC specimens shall be measured in accordance with the same procedures as the substrate specimen. Their resonance frequencies can be detected near the resonance frequency of the substrate specimen as shown in Figure 4.



Key

X frequency

Y intensity

a Shift.

f_s resonance frequency of substrate specimen

f_{TBC} resonance frequency of TBC specimen

Figure 4 — Schematic illustration of the resonance frequencies of substrate and TBC specimens

8 Calculation of Young's modulus

8.1 General

The Young's moduli of the substrate, BC and TC shall be calculated according to 8.2 to 8.4, respectively.

8.2 Young's modulus of the substrate

The Young's modulus of the substrate, E_s, shall be calculated in accordance with ISO 17561:2016, 8.1, Formula (1), on the basis of the dimension, mass and resonance frequency of the substrate specimen, as shown by Formula (1):

$$E_s = 0{,}946\,5 \frac{m_s f_s^2}{b_s} \left(\frac{L_s}{h_s} \right)^3 \left[1 + 6{,}585 \left(\frac{h_s}{L_s} \right)^2 \right] \tag{1}$$

where

f_s is the resonance frequency of the substrate specimen (Hz);

m_s is the mass of the substrate specimen (kg);

L_s is the total length of the substrate specimen (m);

b_s is the width of the substrate specimen (m);

h_s is the substrate thickness (m).

8.3 Young's modulus of BC

The Young's modulus of BC, E_b, shall be calculated in accordance with Formula (2) on the basis of the dimension, mass and resonance frequency of the BC specimen, and the E_s obtained in Formula (1):

$$E_b = \frac{-B + \sqrt{B^2 - 4AC}}{2A} \tag{2}$$

where

$$A = \beta^4 {h_b}^4$$

$$B = 2h_b \left[\beta^4 \left[E_s h_s \left\{ 2{h_b}^2 + 3h_b h_s + 2{h_s}^2 \right\} \right] - 24\pi^2 {f_{BC}}^2 m_{BC} {L_{BC}}^3 / b_{BC} \right]$$

$$C = \beta^4 {E_s}^2 {h_s}^4 - 48\pi^2 {f_{BC}}^2 m_{BC} {L_{BC}}^3 E_s h_s / b_{BC}$$

$$\beta = 4{,}730$$

where

f_{BC} is the resonance frequency of the BC specimen (Hz);

m_{BC} is the mass of the BC specimen (kg);

L_{BC} is the total length of the BC specimen (m);

b_{BC} is the width of the BC specimen(m);

h_b is the BC thickness (m).

8.4 Young's modulus of TC

The Young's modulus of TC, E_c, shall be calculated in accordance with Formula (3) on the basis of the dimension, mass and resonance frequency of the TBC specimen, and the E_s and E_b obtained in Formulae (1) and (2), respectively:

$$E_c = \frac{-B+\sqrt{B^2-4AC}}{2A} \tag{3}$$

where

$$A = \beta^4 h_c^{\,4}$$

$$B = 2h_c\left[\beta^4\left[E_b h_b\left(2h_c^{\,2}+3h_c h_b+2h_b^{\,2}\right)+E_s h_s\left\{2h_c^{\,2}+3h_c h_s+6h_b^{\,2}+6h_b\left(h_c+h_s\right)+2h_s^{\,2}\right\}\right]\right.$$
$$\left.-24\pi^2 f_{TBC}^{\,2} m_{TBC} L_{TBC}^{\,3}/b_{TBC}\right]$$

$$C = \beta^4\left\{E_b^{\,2} h_b^{\,4}+2E_b E_s h_b h_s\left(2h_b^{\,2}+3h_b h_s+2h_s^{\,2}\right)+E_s^{\,2} h_s^{\,4}\right\}-48\pi^2 f_{TBC}^{\,2} m_{TBC} L_{TBC}^{\,3}\left(E_b h_b+E_s h_s\right)/b_{TBC}$$

$$\beta = 4{,}730$$

where

f_{TBC} is the resonance frequency of the TBC specimen (Hz);

m_{TBC} is the mass of the TBC specimen (kg);

L_{TBC} is the total length of the TBC specimen (m);

b_{TBC} is the width of the TBC specimen (m);

h_c is the TC thickness (m).

9 Test report

The test report shall contain the following items:

a) the document used, i.e. ISO 23486;

b) specimen:

   1) material of substrate;

   2) materials and spraying conditions of BC and TC;

   3) dimensions and mass of the substrate, BC and TBC specimens;

   4) thickness of the substrate, BC and TC;

   5) preheat treatment condition (temperature and time);

c) measurement conditions:

   1) type of measuring apparatus;

   2) test temperature and holding time at test temperature;

d) results:

   1) resonance frequencies at test temperature of the substrate, BC and TBC specimens;

2) Young's moduli at test temperature of the substrate, BC and TC;

3) deviations from the procedure and unusual features observed, if any;

4) the date of the measurement.