
**Metallic and other inorganic
coatings — Determination of thermal
conductivity of thermal barrier
coatings at elevated temperature**

*Revêtements métalliques et autres revêtements inorganiques —
Détermination de la conductivité thermique des revêtements
barrières thermiques à température élevée*

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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 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 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 that 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 thermal conductivity is an important property of thermal barrier coatings, ISO 18555 only describes a method for measuring this parameter of thermal barrier coatings at room temperature.

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Metallic and other inorganic coatings — Determination of thermal conductivity of thermal barrier coatings at elevated temperature

1 Scope

This document specifies a method for determining the thermal conductivity of ceramic top coat (TC) constituting thermal barrier coating (TBC) subjected to heat treatment, in a direction normal to the coating surface, 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 14188, *Metallic and other inorganic coatings — Test methods for measuring thermal cycle resistance and thermal shock resistance for thermal barrier coatings*

ISO 18555, *Metallic and other inorganic coatings — Determination of thermal conductivity of thermal barrier coatings*

ISO 18755, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of thermal diffusivity of monolithic ceramics by laser flash method*

EN 821-3, *Advanced technical ceramics – Monolithic ceramics. Thermophysical properties – Part 3: Determination of specific heat capacity*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 14188, ISO 18555 and the following apply.

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

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

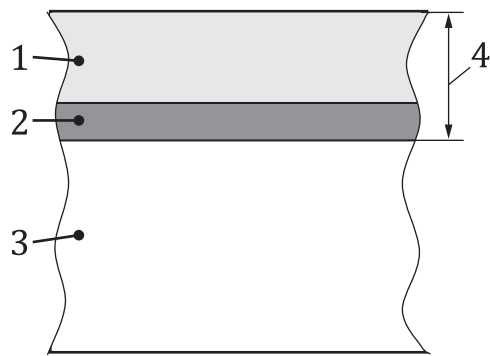
3.1

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 of the top coat through the coating to the substrate of a heat-resistant metallic material

Note 1 to entry: See [Figure 1](#).



- Key**
- | | | | |
|---|----------------|---|-------------------------|
| 1 | top coat (TC) | 3 | substrate |
| 2 | bond coat (BC) | 4 | thermal barrier coating |

Figure 1 — Diagrammatic view of a section of a TBC

4 Principle

The TBC specimen shall be heat-treated prior to the measurement at elevated temperature in order to minimize the change of the coating microstructure. Since the heat-treated BC specimen consisting of the substrate and the BC can be treated as the same as the substrate specimen, the TBC specimen can be regarded as a two-layer model consisting of the substrate and the TC. Thermal conductivities of the substrate and TC are determined in accordance with calculation using the thermal diffusivities, specific heat capacities, and bulk densities^[1]. The fundamental procedures are shown in Figure 2.

The fundamental procedures for determining the thermal diffusivities of the substrate and TC consist of the measurement of temperature-rise curves of two types of specimens (substrate and substrate with TBC) by a flash method, and of calculations. The thermal diffusivity of the TC is obtained by applying a multi-layer analytical model to the temperature-rise curve.

The specific heat capacities and bulk densities of the substrate and TC are separately measured.

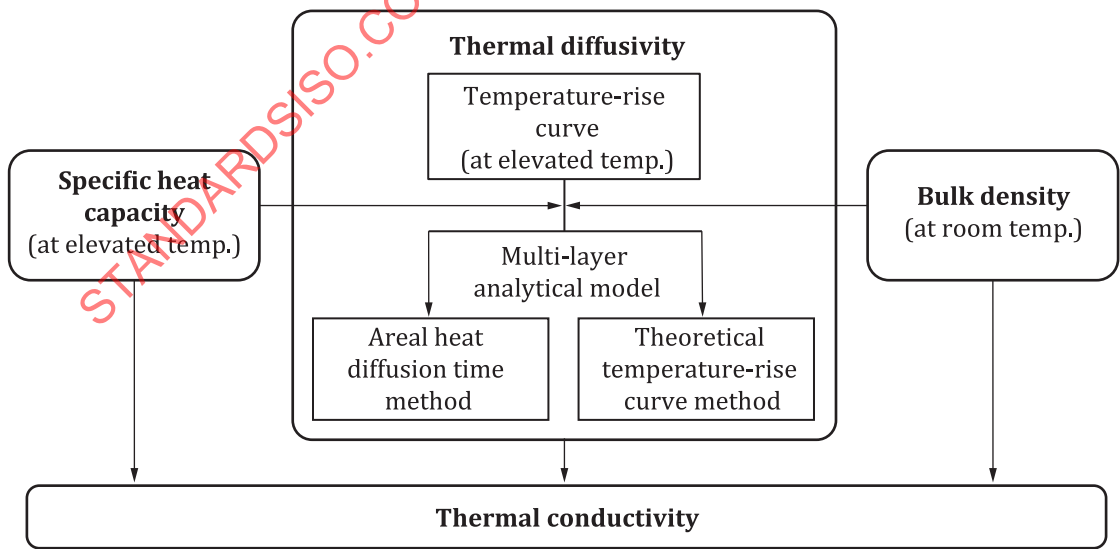
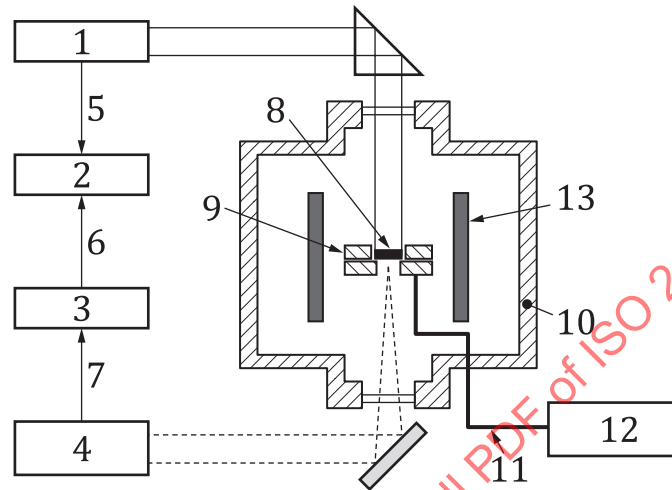


Figure 2 — Fundamental procedures for determining thermal conductivity

5 Apparatus for measuring thermal diffusivity

An example of the apparatus for measuring the thermal diffusivity is schematically shown in [Figure 3](#).

The apparatus consists of pulse heating light source, data recorder, measurement circuit, infrared radiometer, specimen holder, chamber, thermocouple, temperature indicator and heater. The apparatus shall be specified in accordance with ISO 18755 and should be calibrated using reference data and reference materials in reference to ISO 18755:2005, Annex E.



Key

- | | | | |
|---|---|----|-----------------------|
| 1 | pulse heating light source | 8 | specimen |
| 2 | data recorder | 9 | specimen holder |
| 3 | measurement circuit | 10 | chamber |
| 4 | infrared radiometer | 11 | thermocouple |
| 5 | trigger signal | 12 | temperature indicator |
| 6 | amplification of signal | 13 | heater |
| 7 | temperature signal of specimen rear surface | | |

Figure 3 — Typical apparatus for measuring the thermal diffusivity in accordance with the flash method

6 Specimen

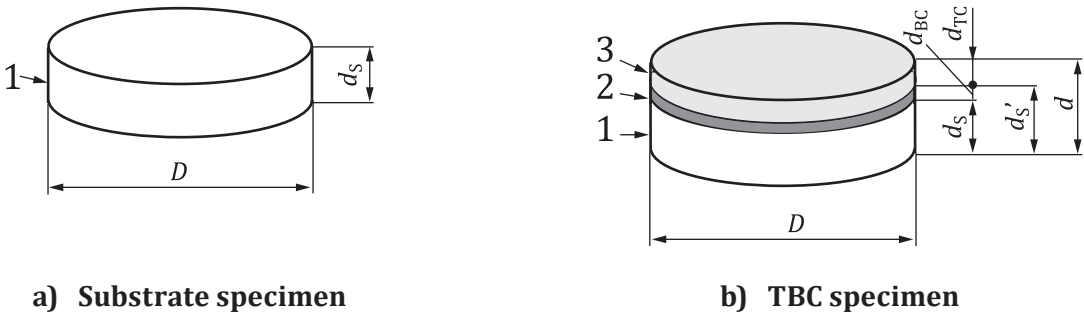
6.1 Shape and dimensions

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

The two types of specimens, the substrate and TBC specimens, shall be used.

The specimen shape shall be a flat disk ([Figure 4](#)) or flat square plate ([Figure 5](#)). The diameter or side length of the specimen shall be from 10×10^{-3} m to 15×10^{-3} m.

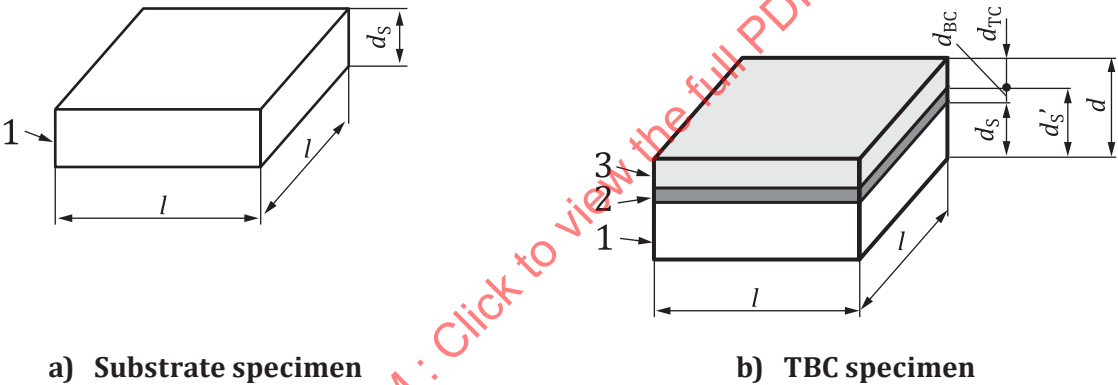
The thickness of substrate, BC, and TC is given in [Table 1](#).



Key

- 1 substrate
- 2 bond coat (BC)
- 3 top coat (TC)
- D diameter
- d_s thickness of substrate
- d_{BC} thickness of BC
- d_{TC} thickness of TC
- d_s' thickness of substrate and BC
- d total thickness of TBC specimen

Figure 4 — Shape of flat disk specimens



Key

- 1 substrate
- 2 bond coat (BC)
- 3 top coat (TC)
- l side length
- d_s thickness of substrate
- d_{BC} thickness of BC
- d_{TC} thickness of TC
- d_s' thickness of substrate and BC
- d total thickness of TBC specimen

Figure 5 — Shape of flat square plate specimens

Table 1 — Thickness of substrate, BC, and TC

Symbol	Designation	Thickness (x 10 ⁻³ m)
d_s	thickness of substrate	$1,00 \leq d_s \leq 2,00$
d_{TC}	thickness of TC	$0,30 (d_s + d_{BC}) \leq d_{TC}$
d	thickness of TBC specimen	$d = d_s + d_{BC} + d_{TC} \leq 3,00$

The thickness tolerance of substrate shall be $\pm 0,01 \times 10^{-3}$ m.

The difference between maximum and minimum thickness shall be $\leq 0,01 d$ for the TBC specimen.

The selections of the shape, the dimension, and the thickness shall be decided in accordance with the agreement between parties involved in the transaction.

6.2 Specimen preparation

The procedure is shown in Figure 6. The TBC specimen preparation shall be as follows.

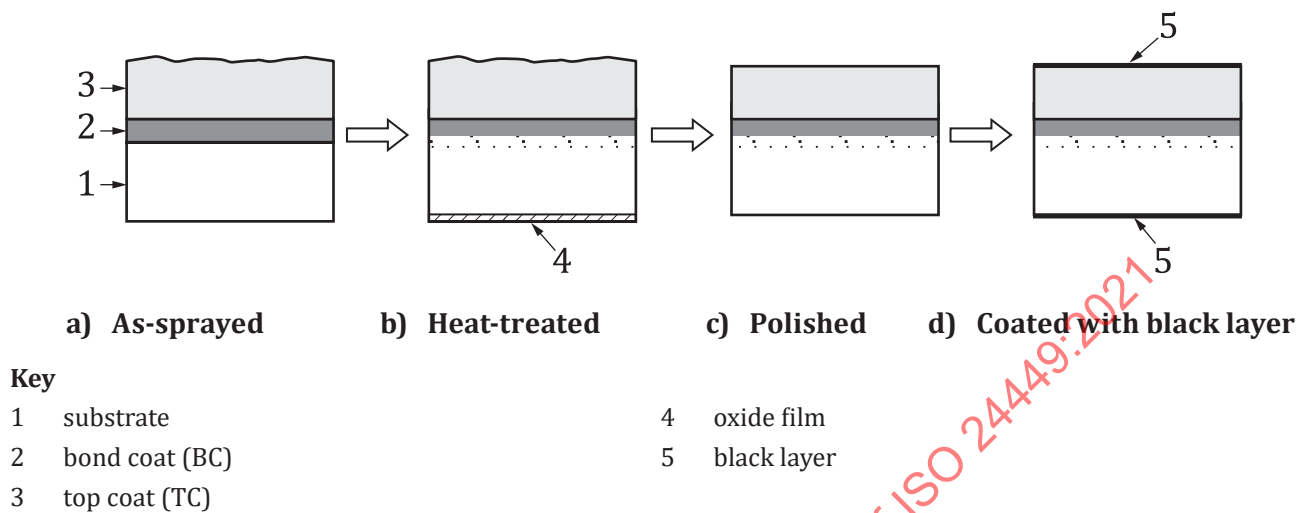


Figure 6 — Procedure of TBC specimen preparation

- TBC specimen shall be heat-treated at the maximum specified test temperature for 2 h to 3 h in air prior to the measurement in order to minimize the change of coating microstructure during the measurement, since the thermal diffusivity and the thermal conductivity of as-sprayed TC change remarkably depending on temperature history.
- The TC surface shall be polished mechanically using abrasive paper P800 or higher in order to smooth the coating surface for the TBC specimen.
- The substrate surface shall be polished mechanically in order to remove the oxide film for the TBC specimen.
- Both surfaces of specimens for measuring thermal diffusivity shall be coated with a thin, opaque, black layer to ensure a high absorption coefficient to the pulse heating light and a high emissivity to the infrared radiometer. Sprayed colloidal graphite is suitable as the black layer. For the surface of the TC, the graphite should be sprayed directly onto it or after sputtering a metal such as gold or platinum onto it.

7 Measuring procedure

7.1 Specimen thickness

The specimen thickness shall be measured at room temperature as follows.

- The specimen thickness shall be measured in accordance with ISO 18755.
- The thickness of TC shall be measured on the image of the coating cross section in accordance with ISO 1463.

7.2 Thermal diffusivity

7.2.1 Measurement of temperature-rise curve

The temperature-rise curve shall be measured as follows.

- The specimen shall be placed in the specimen holder into the chamber. The TBC specimens shall be so fixed that their substrate surfaces are heated by pulse light and the temperatures at the TC surface shall be detected.
- The atmosphere shall be inert gas or vacuum. The selection of the atmosphere shall be decided in accordance with the agreement between parties involved in the transaction.
- The temperature-rise curve shall be measured at room temperature in accordance with ISO 18555.
- The specimen shall be heated to a specified temperature and held for at least 15 min to stabilize the temperature of the specimen.
- The temperature-rise curve shall be measured at the specified temperature.

7.2.2 Calculation of thermal diffusivity of substrate

The diffusivity of substrate shall be calculated based on the temperature-rise curve of the substrate specimen measured at the specified temperature in accordance with ISO 18755.

7.2.3 Calculation of thermal diffusivity of TC

7.2.3.1 General

The calculation of thermal diffusivity TC shall be made based on the temperature-rise curve and with application of a multi-layer analytical model in accordance with ISO 18555. The areal heat diffusion time method or the theoretical temperature-rise curve method shall be used as the multi-layer analytical model. The model shall be chosen in accordance with the agreement between parties involved in the transaction.

7.2.3.2 Areal heat diffusion time method

See ISO 18555:2016, Annex A.

The thermal diffusivity of TC shall be calculated in accordance with [Formulae \(1\)](#) and [\(2\)](#) on the basis of measurement made using the TBC specimen and the thermal diffusivity of substrate obtained in [7.2.2](#):

$$\alpha_{TC} = d_{TC}^2 / \tau_{TC} \quad (1)$$

where

α_{TC} is the thermal diffusivity of TC (m^2/s);

d_{TC} is the thickness of TC (m);

τ_{TC} is the heat diffusion time of TC (s).

$$\tau_{TC} = \frac{6(c_S \rho_S d_S' + c_{TC} \rho_{TC} d_{TC}) \cdot A_{TBC} - (c_S \rho_S d_S' + 3c_{TC} \rho_{TC} d_{TC}) \cdot (d_S'^2 / \alpha_S)}{3c_S \rho_S d_S' + c_{TC} \rho_{TC} d_{TC}} \quad (2)$$

where

- c_S is the specific heat capacity of substrate [J/(kg·K)];
- ρ_S is the bulk density of substrate (kg/m³);
- α_S is the thermal diffusivity of substrate (m²/s);
- d_S' is the thickness of substrate and BC (m);
- c_{TC} is the specific heat capacity of TC [J/(kg·K)];
- ρ_{TC} is the bulk density of TC (kg/m³);
- A_{TBC} is the areal heat diffusion time of TBC specimen (s).

7.2.3.3 Calculation using theoretical temperature-rise curve method

The thermal diffusivity of TC shall be calculated by applying the theoretical temperature-rise curve based on a multi-layer model analysis to the measured temperature-rise curve.

- a) The theoretical temperature-rise curve of the TBC specimen is given in [Formula \(3\)](#);

$$(T(t)/\Delta T)_{th} = F(d_S', d_{TC}, \alpha_S, \alpha_{TC}, c_S, c_{TC}, \rho_S, \rho_{TC}, t) \quad (3)$$

where

- $(T(t)/\Delta T)_{th}$ is the theoretical temperature-rise curve;
- F is a function;
- t is the time (s).

- b) Input the data except for the thermal diffusivity of TC into [Formula \(3\)](#).
- c) The thermal diffusivity of TC shall be calculated by fitting [Formula \(3\)](#) to the measured temperature-rise curve of the TBC specimen.

7.3 Specific heat capacity

The specific heat capacity of the substrate and the TC shall be determined as follows.

- a) The specific heat capacity shall be measured at the specified temperature in accordance with EN 821-3.
- b) The TC stripped off the substrates should be used as the specimen to measure the specific heat capacity of the TC.
- c) The literature values for the TC produced by the same coating process with the powder of the same composition should be used, if the preparation of the TC stripped off the substrate is difficult.
- d) The value measured for the raw material powders of the TC should be used, when the literature values are not available.

7.4 Bulk density

The substrate and TBC specimens shall be used as follows for determining the bulk density of TC.

- a) Measure the mass of the substrate and TBC specimens at room temperature.

- b) Measure the dimensions of the substrate and TBC specimens by using the micrometer and calculate their volumes at room temperature.
- c) The bulk density of substrate shall be determined using the mass and the dimensions of the substrate specimen.
- d) The bulk density of TC shall be determined in accordance with [Formula \(4\)](#);

$$\rho_{TC} = (\rho_{TBC}d - \rho_s(d - d_{TC}))/d_{TC} \quad (4)$$

where

ρ_{TBC} is the bulk density of the TBC specimen (kg/m³);

d is the thickness of TBC specimen (m).

8 Thermal conductivity of TC

Thermal conductivity of TC, λ_{TC} , shall be determined in accordance with [Formula \(5\)](#);

$$\lambda_{TC} = \alpha_{TC} \cdot c_{TC} \cdot \rho_{TC} \quad (5)$$

9 Report

The report shall contain the following items.

- a) a reference to this document, i.e. ISO 24449:2021;
- b) specimen:
 - 1) material of substrate;
 - 2) materials and coating conditions of BC and TC;
 - 3) shape of the specimen (disk or square plate);
 - 4) diameter or side length of the specimen;
 - 5) thickness of substrate, BC, and TC;
 - 6) heat treatment condition prior to measurement (temperature and time).
- c) measurement conditions;
 - 1) type of the apparatus for measuring the thermal diffusivity (model of the instrument);
 - 2) atmosphere for measuring the temperature-rise curve;
 - 3) coating of the specimen for measuring the temperature-rise curve (coating material, coating procedure);
 - 4) measurement temperature and holding time at measurement temperature;
 - 5) calculation method of the thermal diffusivity;
 - 6) method for measuring the specific heat capacity.
- d) results of measurement and calculation.
 - 1) bulk densities at room temperature of substrate and TC;