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

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



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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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The committee responsible for this document is ISO/TC 107, *Metallic and other inorganic coatings*.

Introduction

Thermal barrier coatings are highly advanced material systems. They are generally applied to surfaces of hot-section components made of nickel or cobalt-based superalloys, such as combustors, blades, 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 thermal conductivity is one of the most important properties of thermal barrier coatings, the existing International Standard (ISO 18755:2005) includes only the method for determining the thermal diffusivity of monolithic ceramics, regarding the heat conduction in thermal barrier coating.

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Metallic and other inorganic coatings — Determination of thermal conductivity of thermal barrier coatings

1 Scope

This International Standard specifies the method for determining the thermal conductivities of thermal barrier coatings consisting of metallic bond coats and ceramic top coats, in a direction normal to the coating surface, at room temperature.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 18755:2005, *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*

ASTM E1269-11, *Standard Test Method for Determining Specific Heat Capacity by Differential Scanning Calorimetry*

3 Terms and definitions

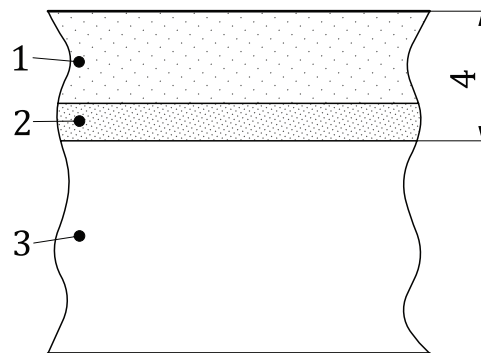
For the purpose of this standard, the terms and definitions given in ISO 18755:2005 and the following apply.

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

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



Key

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

Figure 1 — Diagrammatic view of a section of TBC

[SOURCE: ISO 14188:2012, definition 3.1, modified]

3.2

apparent thermal diffusivity

thermal diffusivity of the specimens [substrate with bond coat (BC) and substrate with thermal barrier coating (TBC)] in a direction normal to the coating surface

3.3

normalized temperature rise

$T(t)/\Delta T$

value which is determined by dividing the difference between the temperature of the specimen rear surface after the pulse heating and the temperature of the specimen rear surface before the pulse heating by the difference between the maximum temperature of the specimen rear surface and the temperature of the specimen rear surface before the pulse heating

$$\frac{T(t)}{\Delta T} = \frac{T_1(t) - T_0}{T_{\max} - T_0}$$

where

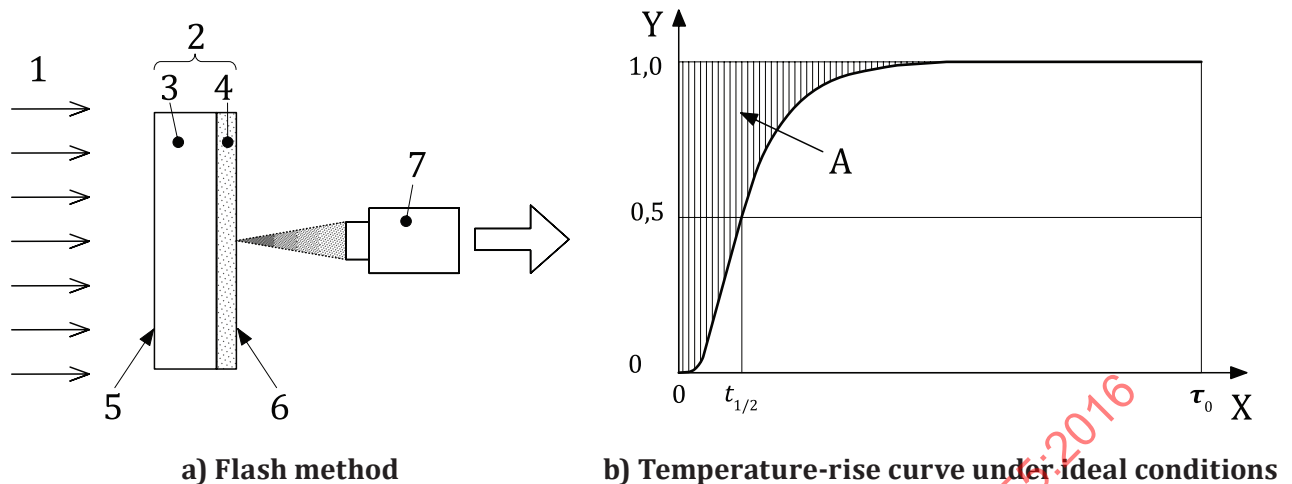
$T_1(t)$ is temperature of specimen rear surface after pulse heating by a flash method;

t is time;

T_0 is temperature of the specimen rear surface before pulse heating;

$T_{\max}$ is maximum temperature of specimen rear surface.

Note 1 to entry: See [Figure 2](#).

**Key**

- 1 pulse heating
- 2 specimen
- 3 substrate
- 4 TBC
- 5 front surface
- 6 rear surface
- 7 infrared radiometer

- X time (s)
- Y normalized temperature rise $T(t) / \Delta T$
- A areal heat diffusion time (s)

Figure 2 — Flash method and temperature-rise curve under ideal conditions

3.4 temperature-rise curve

curve which shows the variation in the normalized temperature rise of the specimen rear surface with time

Note 1 to entry: See the thick solid line in [Figure 2b](#).

3.5 half rise-time

$t_{1/2}$

time required for the normalized temperature rise to reach 0,5 in the temperature-rise curve

Note 1 to entry: See [Figure 2b](#).

3.6 areal heat diffusion time

A

area with time-dimension which is bordered by the horizontal line at the height of the maximum temperature-rise and by the temperature-rise curve

Note 1 to entry: See [Figure 2b](#).

3.7 heat diffusion time

τ_0

time period beginning with pulse heating of the specimen front surface until time at which the specimen temperature becomes uniform

$$\tau_0 = \frac{d^2}{\alpha}$$

where

- τ_0 is heat diffusion time (s);
- d is thickness of specimen (m);
- α is thermal diffusivity (m^2/s).

Note 1 to entry: See [Figure 2b](#).

4 Principle

Thermal conductivities of the substrate, BC, and TC are determined according to calculations using the thermal diffusivities, specific heat capacities and bulk densities. The fundamental procedures are shown in [Figure 3](#).

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

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

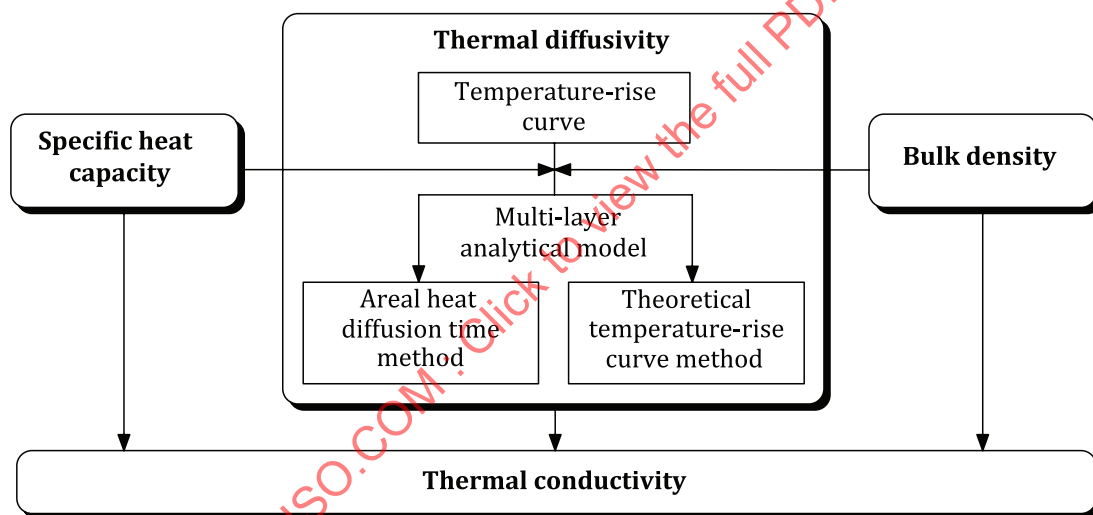


Figure 3 — Fundamental procedures for determining thermal conductivity

5 Apparatus for measuring thermal diffusivity

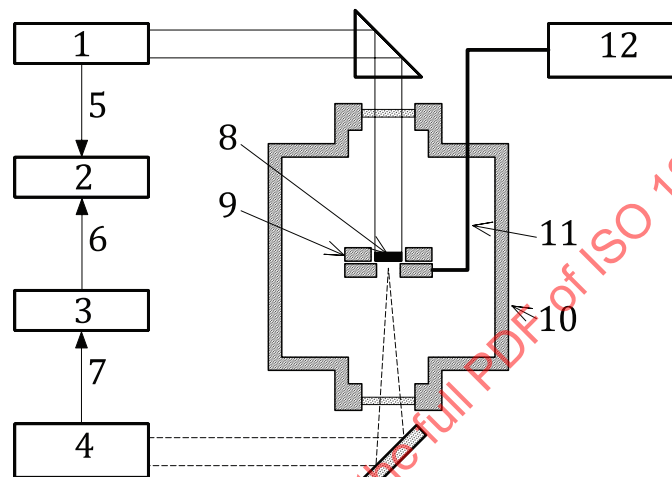
An example of the apparatus for measuring the thermal diffusivity is schematically shown in [Figure 4](#).

The apparatus consists of the following.

- 5.1 Pulse heating light source.
- 5.2 Data recorder.
- 5.3 Measurement circuit.
- 5.4 Infrared radiometer.

5.5 Specimen holder.**5.6 Chamber.****5.7 Thermocouple.****5.8 Temperature indicator.**

The apparatus shall be specified according to ISO 18755:2005 and should be calibrated using reference data and reference materials in reference to Annex E in ISO 18755:2005.

**Key**

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

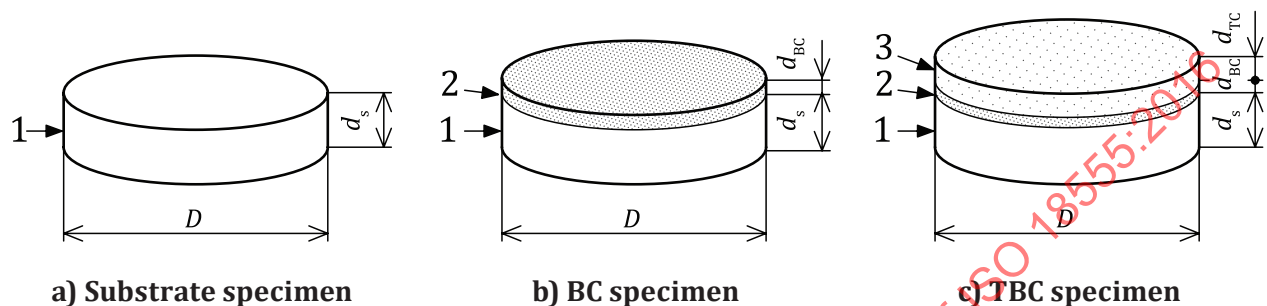
Figure 4 — Typical apparatus for measuring the thermal diffusivity according to a flash method

6 Specimen**6.1 Shape and dimensions**

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

- The three types of specimens (the substrate, BC and TBC specimens) shall be used.
- The specimen shape shall be a flat disk ([Figure 5](#)) or flat square plate ([Figure 6](#)). The diameter or side length of the specimen shall be from 10×10^{-3} m to 15×10^{-3} m.
- The thicknesses of the substrate, BC and TC are given in [Table 1](#).
- The substrate thickness shall be the same for the three types of specimens.
- The thickness tolerance of substrate shall be $\pm 0,01 \times 10^{-3}$ m.
- The thickness of BC shall be the same for the BC and TBC specimens.

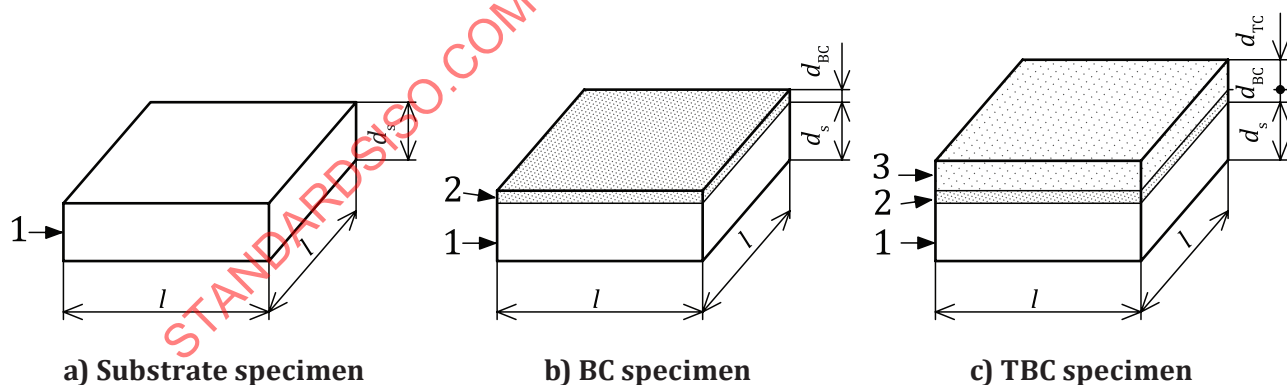
- g) The difference between maximum and minimum thickness shall be $\leq 0,01 d$ for the TBC specimen.
- h) The coating surface should be polished mechanically in order to smooth the coating surface for the BC and TBC specimens.
- i) The selections of the shape, the dimension and the thickness shall be decided according to the agreement between parties involved in the transaction.
- j) For measurement of the specific heat capacities of BC and TC, the coatings stripped off the substrates shall be used as the specimen.



Key

- 1 substrate
- 2 bond coat
- 3 top coat
- D diameter
- d_s thickness of substrate
- d_{BC} thickness of bond coat
- d_{TC} thickness of top coat

Figure 5 — Shape of flat disk specimens



Key

- 1 substrate
- 2 bond coat
- 3 top coat
- l side length
- d_s thickness of substrate
- d_{BC} thickness of bond coat
- d_{TC} thickness of top coat

Figure 6 — Shape of flat square plate specimens

Table 1 — Thicknesses of substrate, BC, and TC

Symbol	Designation	Thickness ($\times 10^{-3}$ m)
d_S	thickness of substrate	$1,00 \leq d_S \leq 2,00$
d_{BC}	thickness of BC	$0,15 \leq d_{BC}$
d_{TC}	thickness of TC	$0,20 (d_S + d_{BC}) \leq d_{TC}$
d	thickness of TBC specimen	$d = d_S + d_{BC} + d_{TC} \leq 3,00$

6.2 Surface treatment

Both surfaces of specimens for measuring thermal diffusivity shall be coated with a thin, opaque, preferably black layer according to ISO 18755:2005.

7 Measuring procedure

7.1 Specimen thickness

The specimen thickness shall be measured as follows.

- The specimen thickness shall be measured according to ISO 18755:2005.
- The thickness of BC and TC shall be measured on the image of the coating cross section according to ISO 1463.

7.2 Thermal diffusivity

7.2.1 Measurement of temperature-rise curve

According to ISO 18755:2005, the temperature-rise curve ([Figure 2b](#)) shall be measured as follows.

- The specimen shall be placed in the specimen holder of the chamber. The BC and TBC specimens shall be so fixed that their substrate surfaces are heated by pulse light and the temperatures at BC and TC surfaces shall be detected (See [Figure 2a](#)).
- The atmosphere is decided according to the agreement between parties involved in the transaction.
- The temperature of the specimen rear surface before pulse heating, T_0 , shall be measured with the thermocouple.
- With variation of the specimen temperature minimized (0,2 K or less per minute) before pulse heating and with the output of infrared radiometer stabilized; the specimen is subject to pulse heating to measure the temperature rise.

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 according to ISO 18755:2005.

7.2.3 Calculation of thermal diffusivities of BC and TC

The calculation of thermal diffusivities of BC and TC shall be made based on the temperature-rise curve and with application of a multi-layer analytical model. 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 according to the agreement between parties involved in the transaction.

7.2.3.1 The areal heat diffusion time method (See Annex A)

a) Calculation of the areal heat diffusion time across the specimen.

The areal heat diffusion time shall be calculated as follows.

Either of the following 1) or 2) may be used.

1) Calculation using numerical data of temperature-rise curve. The areal heat diffusion time, A (hatched portion of Figure 2b) shall be determined directly from the data of temperature-rise curve.

2) Calculation using apparent thermal diffusivity. Using the apparent thermal diffusivity, α_{app} , the areal heat diffusion time, A shall be calculated according to Formula (1).

$$A = d^2 / (6\alpha_{app}) \quad (1)$$

where

d is thickness of specimen (m);

α_{app} is apparent thermal diffusivity of specimen (m²/s).

The apparent thermal diffusivity of the specimen, α_{app} , shall be calculated according to equiareal method or half-time method (see ISO 18755:2005).

3) The correction factors for heat loss and non-uniform heating should be calculated in reference to Annexes B and D respectively in ISO 18755:2005.

b) Calculation of thermal diffusivities of substrate, BC, and TC.

1) The thermal diffusivity of BC shall be calculated according to Formulae (2) and (3) on the basis of the measurement made using the BC specimen, and the thermal diffusivity of substrate obtained in 7.2.2.

The thermal diffusivity of BC can be expressed as follows:

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

where

α_{BC} is thermal diffusivity of BC (m²/s);

d_{BC} is thickness of BC (m);

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

$$\tau_{BC} = \frac{6(c_s \rho_s d_s + c_{BC} \rho_{BC} d_{BC}) A_{BC-S} - (c_s \rho_s d_s + 3c_{BC} \rho_{BC} d_{BC}) (d_s^2 / \alpha_s)}{3c_s \rho_s d_s + c_{BC} \rho_{BC} d_{BC}} \quad (3)$$

where

c_s is specific heat capacity of substrate [J/(kg·K)];

ρ_s is bulk density of substrate (kg/m³);

α_s is thermal diffusivity of substrate (m²/s);

d_s is thickness of substrate (m);

c_{BC} is specific heat capacity of BC [J/(kg·K)];

ρ_{BC} is bulk density of BC (kg/m³);

A_{BC-S} is areal heat diffusion time of BC specimen (s).

2) The thermal diffusivity of TC shall be calculated according to Formulae (4) and (5) on the basis of the measurement made using the TBC specimen, α_s obtained in 7.2.2 and α_{BC} obtained in Formula (2).

The thermal diffusivity of TC can be expressed as follows:

$$\alpha_{TC} = d_{TC}^2 / \tau_{TC} \quad (4)$$

where

α_{TC} is thermal diffusivity of TC (m²/s);

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

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

$$\tau_{TC} = \frac{1}{3c_s\rho_s d_s + 3c_{BC}\rho_{BC}d_{BC} + c_{TC}\rho_{TC}d_{TC}} \left[\begin{aligned} &6(c_s\rho_s d_s + c_{BC}\rho_{BC}d_{BC} + c_{TC}\rho_{TC}d_{TC})A_{TBC-S} \\ &- \left(3c_s\rho_s d_s + c_{BC}\rho_{BC}d_{BC} + 3c_{TC}\rho_{TC}d_{TC} + \frac{6c_s\rho_s d_s c_{TC}\rho_{TC}d_{TC}}{c_{BC}\rho_{BC}d_{BC}} \right) \frac{d_{BC}^2}{\alpha_{BC}} \\ &- (c_s\rho_s d_s + 3c_{BC}\rho_{BC}d_{BC} + 3c_{TC}\rho_{TC}d_{TC}) \frac{d_s^2}{\alpha_s} \end{aligned} \right] \quad (5)$$

where

c_{TC} is specific heat capacity of TC [J/(kg·K)];

ρ_{TC} is bulk density of TC (kg/m³);

A_{TBC-S} is areal heat diffusion time of TBC specimen (s).

7.2.3.2 Calculation using theoretical temperature-rise curve method

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

The theoretical temperature-rise curve shall be chosen according to the agreement between parties involved in the transaction. Examples of the theoretical temperature-rise curve are given in Annex B. The measured temperature-rise curve should be corrected for heat loss and non-uniform heating in reference to ISO 18755:2005 (Annexes B and D respectively).

a) Calculation of thermal diffusivity of BC.

1) The theoretical temperature-rise curve of the BC specimen is given in Formula (6).

$$(T(t) / \Delta T)_{th} = F(d_S, d_{BC}, \alpha_S, \alpha_{BC}, c_S, c_{BC}, \rho_S, \rho_{BC}, t) \quad (6)$$

where

$(T(t)/\Delta T)_{th}$ is theoretical temperature-rise curve;

F is a function.

2) Input the data except for the thermal diffusivity of BC into this equation.

3) The thermal diffusivity of BC shall be calculated by fitting this equation to the measured temperature-rise curve of the BC specimen.

b) Calculation of thermal diffusivity of TC.

1) The theoretical temperature-rise curve of the TBC specimen is given in Formula (7).

$$(T(t) / \Delta T)_{th} = G(d_S, d_{BC}, d_{TC}, \alpha_S, \alpha_{BC}, \alpha_{TC}, c_S, c_{BC}, c_{TC}, \rho_S, \rho_{BC}, \rho_{TC}, t) \quad (7)$$

where G is a function.

2) Input the data except for the thermal diffusivity of TC into this equation.

3) The thermal diffusivity of TC shall be calculated by fitting this equation to the measured temperature-rise curve of the TBC specimen.

7.3 Specific heat capacity

The specific heat capacity shall be determined as follows.

- a) The measurement of the specific heat capacity shall be according to EN 821-3:2005 or ASTM E1269 11.
- b) The literature values for the coating produced by the same coating process with the powder of the same composition should be used, if the preparation of the coating stripped off the substrate is difficult.

NOTE The value measured for the raw material powders of the respective coating can be used, when the literature values are not available.

7.4 Bulk density

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

- a) Measure the mass of the substrate, BC and TBC specimens.
- b) Measure the dimensions of the substrate, BC and TBC specimens by using the micrometer and calculate their volumes.
- c) The bulk density of substrate shall be determined using the mass and the dimensions of the substrate specimen.
- d) The bulk density of BC shall be determined according to Formula (8).

$$\rho_{BC} = (\rho_{BC-S}(d_S + d_{BC}) - \rho_S d_S) / d_{BC} \quad (8)$$

where ρ_{BC-S} is bulk density of the BC specimen (kg/m³).

- e) The bulk density of TC shall be determined according to Formula (9).

$$\rho_{TC} = \left(\rho_{TBC-S} (d_s + d_{BC} + d_{TC}) - \rho_s d_s - \rho_{BC} d_{BC} \right) / d_{TC} \quad (9)$$

where ρ_{TBC-S} is bulk density of the TBC specimen (kg/m³).

8 Thermal conductivities of BC and TC

Thermal conductivities of BC, λ_{BC} , and TC, λ_{TC} , shall be determined respectively by Formulae (10) and (11):

$$\lambda_{BC} = \alpha_{BC} c_{BC} \rho_{BC} \quad (10)$$

$$\lambda_{TC} = \alpha_{TC} c_{TC} \rho_{TC} \quad (11)$$

9 Report

9.1 Items to be reported

The report shall contain the following items. When the reported values are cited, they should be described in the report.

- a) Specimen:
 - 1) material of substrate;
 - 2) materials and spraying conditions of BC and TC, including surface preparation of the substrate;
 - 3) shape of the specimen (disk or square plate);
 - 4) diameter or side length of the specimen;
 - 5) thickness of substrate, BC and TC.
- b) 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) surface treatment of the specimen for measuring the temperature-rise curve (coating material, coating procedure);
 - 4) temperature of specimen rear surface before pulse heating, T_0 ;
 - 5) calculation method of the thermal diffusivity;
 - 6) method for measuring the specific heat capacity.
- c) Results of measurement and calculation:
 - 1) bulk densities of substrate, BC and TC;
 - 2) specific heat capacities of substrate, BC and TC;
 - 3) thermal diffusivities of substrate, BC and TC;
 - 4) thermal conductivities of substrate, BC and TC.

9.2 Additional items to be selected for the report

The report may contain additional items. The additional items are selected from the following, according to the agreement between parties involved in the transaction.

- a) year/month/day of measurement and the measurement laboratory;
- b) manufacturer of the measuring system;
- c) details of thermal diffusivity measurement;
 - 1) correction factors used in [7.2.3.1a](#)) and [7.2.3.2](#);
 - 2) type of thermocouple and positional relationship between the thermocouple and specimen;
 - 3) type of the infrared radiometer;
 - 4) pulse heating light source (type, intensity, pulse width, pulse beam centre of gravity and the method to determine the centre of gravity);
 - 5) number of the measurement;
 - 6) data regarding the temperature-rise curve (the representative data in case of necessity);
 - 7) half-rise time or areal heat diffusion time.

Annex A (informative)

Areal heat diffusion time method

A.1 General

A multi-layer model is shown in [Figure A.1](#). According to the multi-layer model, based on the response function method,^[2] the areal heat diffusion time A [s] across the multi-layer specimen is expressed as:

$$A = \int_0^{\infty} \left[1 - b\sqrt{\tau} \cdot T_r(t) \right] dt = \lim_{\xi \rightarrow 0} \left[\frac{1}{\xi} - b\sqrt{\tau} \cdot \tilde{T}_r(\xi) \right] \quad (\text{A.1})$$

where

b is thermal effusivity across the multi-layer specimen;

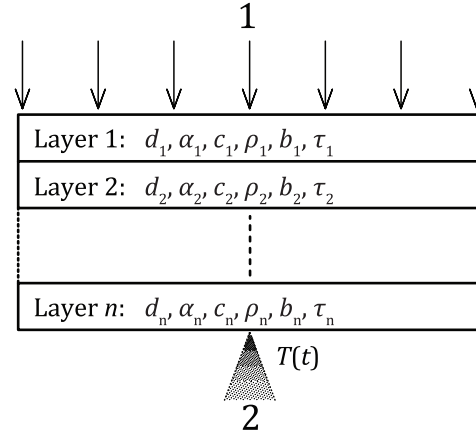
τ is heat diffusion time across the multi-layer specimen;

$T_r(t) = T(t) / \Delta T$ is normalized temperature rise (see [3.3](#));

$\tilde{T}_r(\xi) = \int_0^{\infty} T_r(t) \exp(-\xi t) dt$ is Laplace transform of $T_r(t)$;

ξ is a Laplace parameter.

The heat diffusion across the n -layered specimen is systematically analysed using Formula (A.1).



Key

- 1 pulse heating
- 2 infrared radiometer
- d_i thickness of the i th layer ($i = 1, 2, \dots, n$)
- α_i thermal diffusivity of the i th layer ($i = 1, 2, \dots, n$)
- c_i specific heat capacity of the i th layer ($i = 1, 2, \dots, n$)
- ρ_i bulk density of the i th layer ($i = 1, 2, \dots, n$)
- b_i thermal effusivity of the i th layer ($i = 1, 2, \dots, n$)
- τ_i heat diffusion time of the i th layer ($i = 1, 2, \dots, n$)

Figure A.1 — Multi-layer model

A.2 Single-layer model

The areal heat diffusion time across a single-layer specimen A_{1-s} is expressed as:

$$A_{1-s} = \tau_1 / 6 \quad (A.2)$$

From Formula (A.2), the heat diffusion time of the first layer is determined by Formula (A.3):

$$\tau_1 = 6A_{1-s} \quad (A.3)$$

A.3 Two-layer model

The areal heat diffusion time across a two layer specimen A_{2-s} is expressed as:

$$A_{2-s} = \frac{b_1 \tau_1^{3/2} + 3b_2 \tau_1 \tau_2^{1/2} + 3b_1 \tau_1^{1/2} \tau_2 + b_2 \tau_2^{3/2}}{6(b_1 \sqrt{\tau_1} + b_2 \sqrt{\tau_2})} \quad (A.4)$$

From Formula (A.4), the heat diffusion time of the 2nd layer is determined by Formula (A.5):

$$\tau_2 = \frac{6A_{2-s}(c_1 \rho_1 d_1 + c_2 \rho_2 d_2) - (c_1 \rho_1 d_1 + 3c_2 \rho_2 d_2) \tau_1}{3c_1 \rho_1 d_1 + c_2 \rho_2 d_2} \quad (A.5)$$