INTERNATIONAL STANDARD

ISO
13826

Metallic and other inorganic coatings — Determination of thermal diffusivity of thermally sprayed ceramic coatings by laser flash method

Revêtements métalliques et autres revêtements inorganiques — Détermination de la diffusivité thermique des revêtements céramiques obtenus par projection thermique par la méthode flash laser

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Foreword

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Introduction

Thermally sprayed ceramic coatings provide protection against high-temperature corrosion, erosion and wear; they can also change the appearance, electrical or tribological properties of the surface, replace worn material, etc. Thermal barrier coatings (TBCs) are typical examples of such ceramic coatings.

Thermal diffusivity data of thermally sprayed ceramic coatings are measured by the laser flash method. The data are used to calculate thermal conductivity when provided with density and specific heat capacity data.

Thermal diffusivity and thermal conductivity are significant properties of such thermally sprayed coatings when designing for thermal insulation, thermal isolation, efficient heat transfer and cooling systems. It is used by designers to calculate appropriate thickness needed to protect the metallic components and thus to determine the maximum temperature to which super-alloys with the thermally sprayed ceramic coatings could be exposed.

This International Standard gives guidelines for the determination of thermal diffusivity of thermally sprayed ceramic coatings by the laser flash method.

Metallic and other inorganic coatings — Determination of thermal diffusivity of thermally sprayed ceramic coatings by laser flash method

1 Scope

This International Standard specifies a test method of thermal diffusivity of thermally sprayed ceramic coatings using laser technology.

This test method is applicable to self-standing thermally sprayed ceramic coatings of thickness between 0,8 mm to 2 mm, deposited by various thermal spray processes and removed from the substrate. Thermal diffusivity values ranging from 10^{-7} m^2/s to 10^{-4} m^2/s are measurable by this test method for temperature range from 300 K to 1500 K.

2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

2.1
thermal diffusivity
α
ratio of thermal conductivity to specific heat capacity per unit mass, which describes the rate at which heat flows through a material, expressed in m^2/s

2.2
half-rise time
$t_{1/2}$
time needed for the rear surface temperature to reach one-half of its maximum value or half-rise time, expressed in s

3 Principle of test method

The laser flash method is based on the measurement of the temperature profile of the rear surface of the sample when a pulsed laser illuminates the front surface, thus avoiding interference between the sensor, recording the temperature rise on the rear surface, and heat source.

Thermal diffusivity shall be measured by applying a high-intensity short-duration heat pulse to one face of a parallel-sided homogeneous test part, monitoring the temperature rise at the opposite face as a function of time, and determining the transient half-rise time ($t_{1/2}$).[1]

Thermal diffusivity is calculated as

$$\alpha = \omega \left(\frac{L^2}{t_{1/2}} \right)$$

(1)

where

$\quad \omega \quad$ is the thermal diffusivity correction factor;

$\quad L \quad$ is the thickness of the specimen, m;

4 Test method

4.1 General

The laser flash method determines the thermal diffusivity for coatings on known substrates using small disc-shaped specimens. Covering an application range from 300 K to 1500 K, it fulfils the needs of thermal classification for almost any material and system problem, including ceramic thermal barrier coatings. The laser flash method is currently the most widely accepted method for precise measurement of the thermal diffusivity.[2]

4.2 Apparatus

The essential features of a laser flash diffusivity unit are shown in Figure 1.
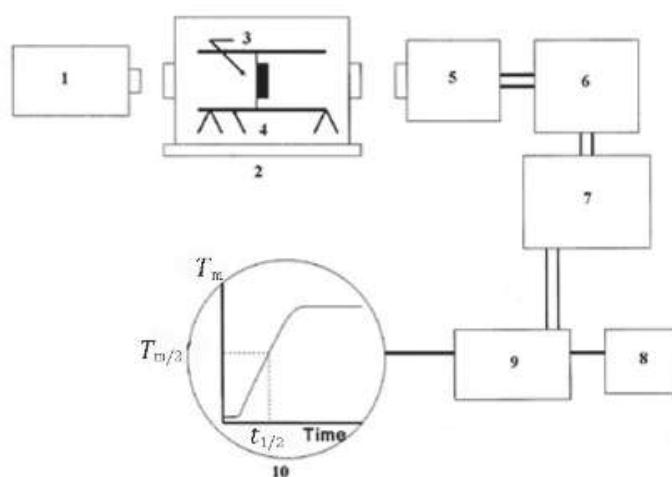
Key

1   Laser
2   Vacuum or inert atmosphere
3   Sample
4   Heater
5   Detector
6   AD-converter and amplifier
7   Digital Data Acquisition System
8   Printer
9   PC
10 Visual Display

Figure 1 — Schematic diagram of laser flash diffusivity unit

The window is of any transparent material and the specimen holder is ceramic or any other material with lower thermal conductivity than the sample.

4.2.1 Flash source

It is essential for the laser source to be uniform over the entire surface of the specimen. For this reason, Neodymium (Nd) glass laser as a flash source is preferred. The pulse width, FWHM (full width at half maximum), should be shorter than 1,0 ms.

4.2.2 Heater

The heater shall be of adequate dimension to heat and fix within the temperature range. It shall be capable of having a suitable temperature control facility prior to and during a test to be less than 4 % of the maximum temperature rise.

4.2.3 Environmental control cabinet

The cabinet, capable of measuring below and above room temperature, may be a vacuum chamber or contain inert gas for operation in a protective atmosphere and is fitted with a window, which is transparent to the flash source. A second window for detection of the rear surface temperature rise shall be transparent. The optical detector shall be shielded from direct exposure to the energy beam with appropriate filters.

4.2.4 Detector and signal conditioning

For the measurement of thermal diffusivity, a non-contact infrared type sensor is preferable. However, detectors may consist of thermocouples, optical pyrometers, infrared types, etc. that will provide a linear electrical output proportional to a small temperature rise, capable of sensing a 0,05 °C change in temperature. The detector and its associated amplifier shall have a response time substantially smaller than 2 % of the 'half-rise time' value.

The signal processor includes the electronic circuit to bias out the ambient temperature reading, spike filters, amplifiers, and analog-to-digital converters.

4.2.5 Data acquisition system

The response frequency of the data acquisition system should preferably be faster than about 1 MHz per channel and should have a resolution of A/D converter more than 12 bit.

5 Test specimen (disc)

The optimum thickness of specimens depends on the magnitude of the thermal diffusivity of the coating. For low thermal diffusivity, the thickness of the specimen is approximately 1 mm; however, for high thermal diffusivity of the coating, a thick sample is used.

The typical specimens for the determination of thermal diffusivity are thin circular discs of 10 mm in diameter and between 0,8 mm to 2 mm in thickness. Faces of specimens shall be flat and parallel within 0,5 % of their thickness.[3] Non-uniformity of surfaces is to be avoided to prevent errors in the measurement of average thicknesses which gives errors in thermal diffusivity data.

For the separation of the coating, either the substrate is prepared with NaCl on the substrate prior to coating by immersing in water to remove the coating, or the coating is applied on a graphite substrate which is then heated to 1000 K for 1 h for removal of the coating.

Mostly, specimens are coated on the front, and at the rear face if an infrared detector is used to check temperature rise, with an opaque or non-reflective film of platinum, nickel, copper, gold or colloidal graphite. These deposits, between 5 μm to 10 μm, shall not react with the specimen or melt or vaporize over a specified temperature range.

6 Procedures

The following preparation procedures of the specimens shall be carried out.

a) Record mass, dimensions and density of test specimen.

b) Mount it in a sample holder, set up a vacuum or inert gas atmosphere.

c) Record the specimen temperature and irradiate the test part with a laser beam. Monitor the base line and transient-rise and cooling data to determine maximum temperature rise, given as ΔT_{mT}, where 'mT' is the maximum temperature, in K. Assess the half time, $t_{1/2}$ value from the initiation of the pulse for the rear-face temperature to reach the half-maximum amount.

d) Calculate the heat loss calculation factor η_h, finite pulse time effect factor, η_f and thermocouple response time-effects correction factor η_c from the temperature rise data (see Annex A for calculation of correction factors).

e) Change the specimen temperature to next value and repeat the tests at different temperatures to obtain appropriate data.

7 Calculation of thermal diffusivity

Following Formula (1) and half-rise time, $t_{1/2}$ from Figure 2, calculate the thermal diffusivity taking into account heat loss correction, finite pulse time-effects correction and thermocouple response time-effects correction given in Annex A.

Key
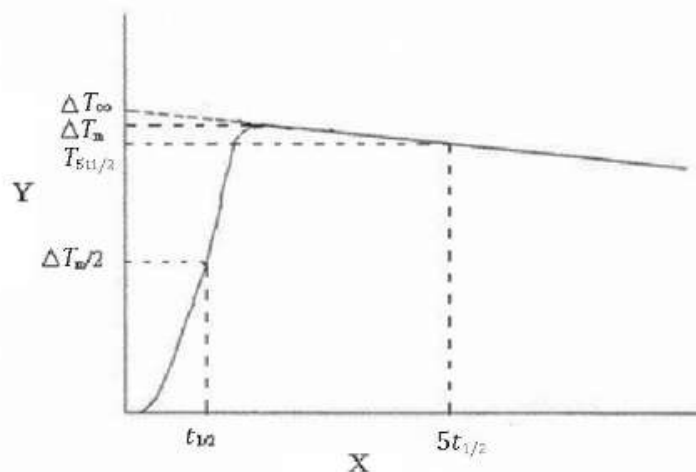
τ pulse time

$t_{1/2}$ half-rise time

ΔT_m temperature difference between line and maximum rise in temperature

Δ_∞ theoretical maximum rise

X   time/s

Y   temperature/K

Figure 2 — Temperature rise as a function of time

Thermal diffusivity is able to be calculated using other analysis methods, for example, half-rise time method without correction and curve fitting method. The user should report in detail if they use another analysis method instead of Annex A.

8 Test report

The test report shall contain following information:

a) a reference to this International Standard;

b) name and address of the testing organization;

c)   date of the test;

d)   description of the material of test specimen;

e)   mass, dimensions of the test specimen, reference sample (sapphire) and glassy carbon;

f)   calculated value of bulk density;

g)   laser pulse source;

h)   environmental conditions;

i)   specimen temperature (°C) and type of temperature rise detector;

j)   recorded value of the maximum temperature rise, ΔT_{mT} and the half-maximum temperature rise time, $t_{1/2}$;

k)   calculated values of thermal diffusivity at each measured temperature;

l)   statistical results of repeated measurements at each temperature;

m)   any additional observations concerning the test.

Annex A
(informative)

Calculations of thermal diffusivity after appropriate corrections

A.1 General

For the measurement of thermal diffusivity from Formula (1),

$$\alpha = \omega \left(\frac{L^2}{t_{1/2}} \right)$$

where

  ω is the thermal diffusivity correction factor;

  L is the thickness of the specimen, m.

the following corrections are required.

A.1.1 Heat loss correction

Heat loss due to vacuum in the control cabinet is primarily due to radiation heat exchange.[4] The relationship between the temperature rise and thermal diffusivity is described using Formula (A.1):

$$\Delta T = \Delta T_\infty \sum_{n=1}^{\infty} Y_n(0) Y_n(L) \exp\left(-\beta_n^2 \frac{\alpha t}{L^2} \right) \tag{A.1}$$

where

$$\Delta T_\infty = \frac{Q}{\rho C_p L} \; ;$$

$$Y_n(x) = \frac{\sqrt{2}\left(\beta_n^2 + H^2\right)^{1/2}\left[\beta_n \cos\left(\frac{\beta_n x}{L} \right) + H \sin\left(\frac{\beta_n x}{L} \right) \right]}{\sqrt{\left(\beta_n^2 + H^2\right)\left(\beta_n^2 + H^2 + H\right) + H\left(\beta_n^2 + H^2\right)}} \; ;$$

$$H = \frac{hL}{k} \; ;$$

$$\left(\beta_n^2 - H^2\right) tg\beta = 2H\beta \; ;$$

$n = 1, 2, 3, 4...$

where

  Q is the heat quantity, J;

  L is the thickness of the specimen, m.

If there is no heat loss, i.e. $h = 0$, Formula (A.1) can be described as

$$\Delta T = \Delta T_\infty \left[1 + 2 \sum_{n=1}^{\infty} (-1)^n \exp\left(-\frac{n^2 \pi^2 \alpha_0 t}{L^2} \right) \right] \tag{A.2}$$

If there is heat loss ($h > 0$) during the measurement procedure, Formula (A.1) can be plotted as Figure A.1 with $\Delta T_{5t1/2}$, the rear-face temperature rise after $5t_{1/2}$. Figure A.1 shows the relationship between $T_{5t1/2}$ and heat loss correction factor η_h, from which the heat loss correction factor is calculated.

Table A.1 shows heat loss correction factors η_h at different $T_{5t1/2}/T_{t1/2}$.
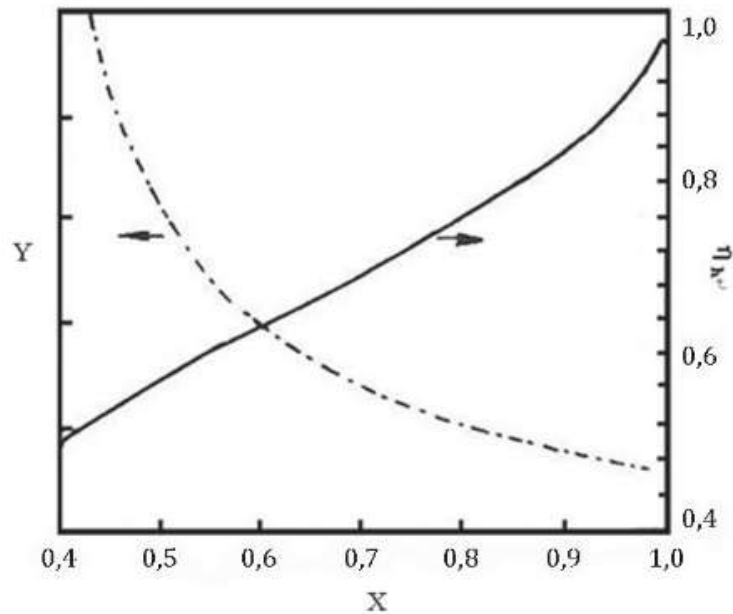
Key
X $\Delta T_{5t1/2}/\Delta T_\infty$
Y $\Delta T/\Delta T_\infty$

Figure A.1 — Heat loss correction factor η_h as a function of ratio $\Delta T_{5t1/2}$ and ΔT_∞

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Table A.1 — Values of the heat loss correction factor η_h at different $T_{5t1/2}/T_{t1/2}$

$T_{5t1/2}/T_{t1/2}$	η_h	$T_{5t1/2}/T_{t1/2}$	η_h
2,00	1	1,85	0,856
1,99	0,957	1,835	0,849
1,98	0,942	1,82	0,842
1,97	0,935	1,80	0,835
1,96	0,928	1,785	0,827
1,95	0,921	1,77	0,820
1,945	0,914	1,755	0,813
1,93	0,906	1,735	0,806
1,92	0,899	1,72	0,799
1,91	0,892	1,70	0,791
1,90	0,885	1,68	0,784
1,89	0,878	1,66	0,777
1,875	0,870	1,64	0,770
1,86	0,863	1,62	0,763

A.1.2   Finite pulse time-effects correction

It is assumed that due to finite pulse effect, measurement of thermal diffusivity will be influenced. The relationship between the finite pulse time-effects correction factors and $t_{1/2}$ is described as:

$$\eta_f = \frac{2,92t_{1/2}}{\tau} \sum_{n=1}^{\infty} \frac{(-1))}{n^2} \exp\left(-1,37n^2\eta_f\right)\left[1-\exp\left(\frac{1,37n^2\tau\eta_f}{t_{1/2}}\right)\right] \tag{A.3}$$

Plotting Formula (A.3) as Figure A.2, pulse time effect η_f can be recorded as a function of ratio of $\tau/t_{1/2}$, where τ is pulse time and $t_{1/2}$ is 'half-rise' time. To minimize the finite pulse time effects, $\tau/t_{1/2}$ shall be less than 0,02. If there is sufficient pulse width $(\tau/t_{1/2})$, η_f is 1,0.
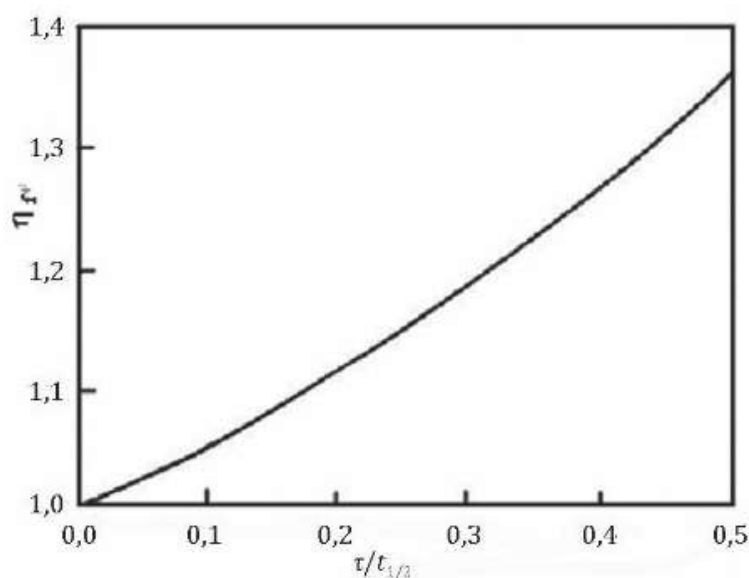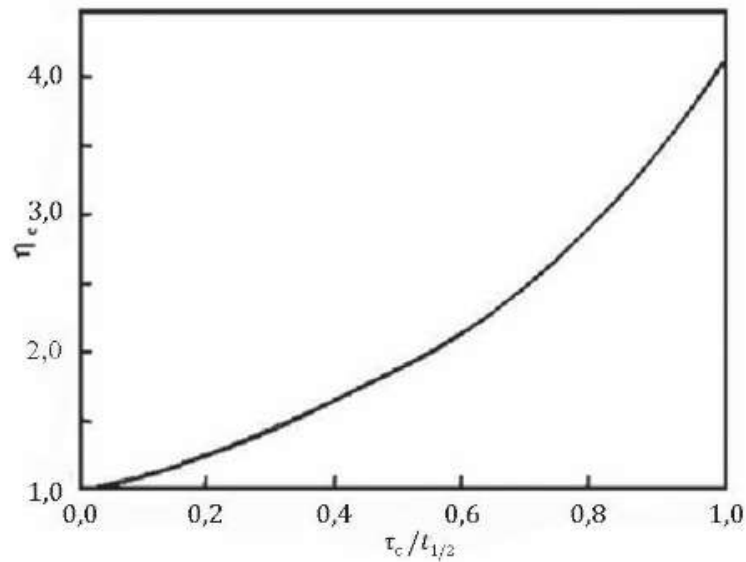

Figure A.2 — Pulse time effect η_f as a function of ratio $\tau/t_{1/2}$

A.1.3 Thermocouple response time-effects correction

Distortion due to the response time effects of the thermocouple, if the measurement system uses the thermocouple as a temperature detector, is shown in <u>Figure A.3</u>. The relationship can be described as:

$$\frac{1}{2} = \exp\left(-\frac{t_{1/2}}{\tau_t}\right) + 2\sum_{n=1}^{\infty} \frac{(-1)^2}{\left(1 - \frac{1,37 n^2 \tau_c \eta_c}{t_{1/2}}\right)} \left[\exp\left(-1,37\eta_c n^2\right) - \exp\left(\frac{t_{1/2}}{\tau_c}\right)\right] \tag{A.4}$$

where

τ_c is the time constant of thermocouple for known material and dimensions;

η_c is the thermocouple response time-effects correction factor.

Figure A.3 — Thermocouple response time-effects correction factor η_c as a function of ratio $\tau_c/t_{1/2}$

The relationship between τ_c and η_c is shown in <u>Figure A.3</u> following the calculation of Formula (A.4). Therefore, to avoid thermocouple response time effects, use of non-contact type sensor for the measurement of thermal diffusivity is supported.

A.1.4 Calculation

Taking into account all the above correction factors, thermal diffusivity α is calculated as follows:

$$\alpha = \frac{0,139\eta_h \eta_f \eta_c L^2}{t_{1/2}} \tag{A.5}$$

where

η_h is the heat loss correction factor;

η_f is the finite pulse time-effects correction factor;

η_c is the thermocouple response time-effects correction factor;

L is the specimen thickness, m;

$t_{1/2}$ is the half-rise time, s.

The test method is considered as an absolute method of measurement because no reference sample is required. For verification of performance of the instrument system, use of reference materials are required.

Bibliography

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

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[3]   ASTM E1461, *Standard Test Method for Thermal Diffusivity by the Flash Method*

[4]   YUQIN G., & HONG L. Analysis and modification of thermophysical property measurements by laser pulse method. *Journal of Astronautic Metrology and Measurement.* 1977, **7** pp. 42–48

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