
**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

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 13826 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*.

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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.

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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} m²/s to 10^{-4} m²/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 m²/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

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

where

ω is the thermal diffusivity correction factor;

L is the thickness of the specimen, m;

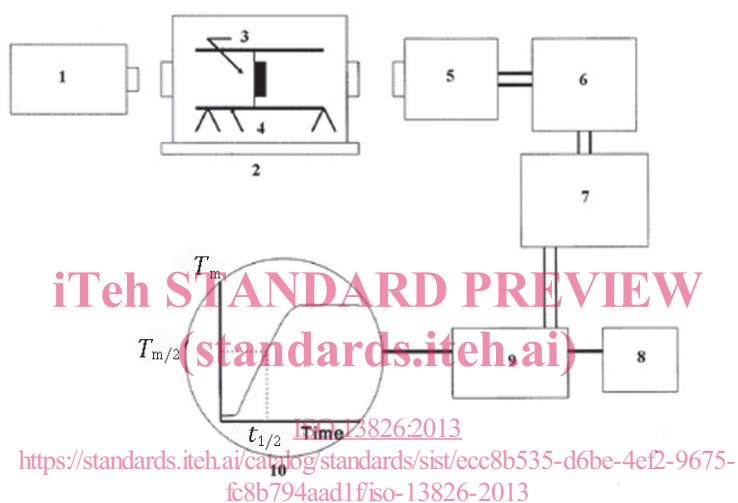
4 Test method

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.

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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.