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Fine ceramics (advanced ceramics, advanced technical ceramics) — Thermophysical properties of ceramic composites — Determination of unidimensional thermal diffusivity by flash method

Céramiques techniques — Propriétés thermophysiques des composites céramiques — Détermination de la diffusivité thermique unidimensionnelle par la méthode flash

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Foreword

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This document was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

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Fine ceramics (advanced ceramics, advanced technical ceramics) — Thermophysical properties of ceramic composites — Determination of unidimensional thermal diffusivity by flash method

1 Scope

This document describes the flash method for the determination of thermal diffusivity of ceramic matrix composites with continuous fibre reinforcement.

In order to conform with the unidimensional heat transfer hypothesis, the experimental conditions are defined such that the material behaves in a homogeneous manner. This involves performing tests in one symmetry axis of the composite.

The method is applicable to materials which are physically and chemically stable during the measurement, and covers the range of temperature from 100 K to 2 800 K. It is suitable for the measurement of thermal diffusivity values in the range 10^{-4} m^2·s^{-1} to 10^{-7} m^2·s^{-1}.

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

ISO 20507, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Vocabulary*

EN 60584-1, *Thermocouples — Part 1: Reference tables (IEC 60584-1:1995)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 20507 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 diffusivity
a
ratio of the thermal conductivity to the product of the bulk density and the specific heat capacity

3.2
transient half time
$t_{1/2}$
time from the initiation of the pulse until the increase of the temperature on the back face of the test specimen reaches one half of the maximum temperature increase

3.3

thickness

h

dimension of the test specimen in the direction of heat transfer measurement

4 Principle

One side of a plane- and parallel-face test piece is exposed to a uniformly distributed energy pulse that is of very short duration compared with the transient half time.

The transient temperature rise (ΔT) on the opposite face (back face) or a quantity directly proportional to ΔT is recorded as a function of time (t) (see Figure 1).

The thermal diffusivity is obtained by comparing the experimental thermogram with a theoretical model, which is a unidimensional analytical thermal model, with two parameters, as described in Annex A. If other models are used, they are to be specified in the test report.

5 Apparatus

5.1 Heat pulse source

The heat pulse source may be a flash tube or a pulse laser.

The pulse energy shall be as uniform as possible over the front face of the test piece.

5.2 Test chamber

The test chamber shall be either a furnace or a cryostat, capable of operation within the temperature range required, or a draught proof enclosure for ambient temperature measurement.

The design of the furnace shall meet the following requirements:

a) it shall contain a working area in which the spatial temperature gradient is sufficiently low (≤5 K over working area width) to result in a homogeneous temperature on the test piece;

b) in steady state conditions, the drift in temperature shall be less than 0,01 K/s;

c) the heat pulse source may be placed either inside the furnace or outside the furnace; in the latter case, the furnace shall be fitted with a window, transparent to the pulse radiation;

d) the furnace shall provide suitable access for measurement of ΔT or a quantity directly proportional to ΔT on the back face of the test piece.

When the test is performed under gas, the test piece should be in a horizontal position in order to reduce convection effects of the gas on the specimen.

NOTE Measurement under vacuum will reduce convection losses and will limit the oxidation phenomena at high temperature.

5.3 Detectors

5.3.1 Measurement of absolute temperature

The temperature of the test piece shall be measured either with a thermocouple (in accordance with EN 60584-1) or with an optical pyrometer.

5.3.2 Transient detectors

The detector shall be either a quantum radiation detector, a thermocouple or any other means that does not disturb the measurement of the transient response of the specimen. It shall be capable of detecting changes of 0,05 K in the temperature of the test piece, with a linear response over the range of temperature change less than or equal to 5 K.

The choice of the detector depends primarily on both the temperature range in which the measurement is to be performed and the characteristic time of the sample. The first parameter sets the spectral range for which the detection sensitivity of the detector shall be maximum; the second parameter fixes its time constant.

It shall have a response time as shown in Formula (1).

$$t_d \leq 0{,}002\ h^2/a \tag{1}$$

where

t_d is the response time, in seconds (s);

h is the thickness, in metres (m);

a is the thermal diffusivity, in square metres per second ($m^2 \cdot s^{-1}$).

This condition shall be verified afterwards and, if it is not met, the size of the specimen shall be increased.

The infrared detector, when used, shall be of a type appropriate to the minimum test piece temperature, for example:

a) HgCdTe or PbSnTe cells, liquid nitrogen cooled, for test specimen temperatures within the range 300 K to 800 K;

b) PbS or InSb cells for test specimen temperatures above 500 K.

Care shall be taken that the signal comes only from the central area of the back face, that is with a tolerance of 5 % of the diameter of the test specimen.

When used, thermocouples shall be of the separated junction type, the hot junction being the back face of the test piece. They shall be in accordance with EN 60584-1. Electrically non-conductive material shall be coated on the front face and on the rear face, with a thin coating of high thermal conductivity material in order to ensure accurate measurement of surface temperatures.

In order to minimize heat losses, the use of the thermocouples with wires of the smallest possible diameter is recommended.

The thermocouple type most often used is chromel-alumel for measurements from room temperature up to 1 100 K. Semi-conducting couples may also be used: Bi_2Te_3 from 90 K to 400 K and $FeSi_2$ for temperatures up to 1 100 K. For temperatures over 1 100 K, a non-contact measurement technique is recommended.

5.4 Data acquisition

The data acquisition system used may be analogue or digital. It shall be equipped with a means of recording the temperature change versus time (before, during and after the pulse) and the time origin. These means shall be accurate to within 0,02 ms.

6 Test specimens

The size of the test specimens shall be fixed to meet the requirements for application of the chosen thermal model (for example like the one described in Annex A). Generally a disc of a diameter between 1 to 8 mm is used.

The thickness of the test specimen shall be defined in relation to the characteristics of the heat pulse source to minimize the uncertainty factors coming from the poorly controlled boundary conditions such as (i) the pulse duration, (ii) the non-uniformity of the incident flux, (iii) the heat losses and (iv) the nonlinearity (see diagram reported in Annex B).

7 Test specimen preparation

7.1 Machining and preparation

Test specimens shall be cut with their longitudinal axis coinciding with one of the principal directions of the reinforcement. The faces perpendicular to the measurement face shall be flat and parallel. The parallelism error of the two faces shall be less than 0,05 mm.

If the test specimen is transparent to the infrared radiation at the considered wavelength of the heat source, a coating is necessary. This coating shall be opaque, absorbent, adherent and compatible with the test specimen. The thickness of the coating shall be as thin as possible in order to not influence the diffusivity measurement.

If the test specimen is non-conductive, and if a thermocouple is used to measure the temperature on the back face, an adequate conductive coating shall be used.

7.2 Number of test specimens

A minimum of three test specimens shall be tested.

8 Procedure

8.1 Calibration of apparatus

As the measurement of thermal diffusivity is an absolute method, reference type materials with known diffusivities can be used to check the system. The homogeneity of the laser beam can be verified by use of a photocell or a photodiode.

NOTE There is no recognized standard reference material for thermal diffusivity measurements, although several materials are used (for example POCO graphite, ARMCO iron).

8.2 Procedure

The pulse duration shall be less than or equal to 0,002 h^2/a to allow for direct application of the theoretical model. In general this corresponds to a period less than 1/50 of transient half time ($t_{1/2}$).

NOTE When this condition is not obeyed, a correction of the thermogram is possible by placing the time origin at the energetic barycentre t_b of pulse (see Figure 1).

Measure the thickness of the test specimen within 0,01 mm, using micrometer callipers in accordance with ISO 3611. In cases where a coating is used, make the measurement before coating. When the change in thickness due to thermal expansion is larger than 1 %, apply a correction to the measured thickness value.

Fix the test specimen such that the front face is perpendicular to the heat source beam. Thermal losses from the specimen to the surrounding environment shall be kept to a minimum and the contact area of the test piece with the sample holder shall be as small as possible.

After the test specimen has reached a constant temperature, its front face is exposed to the heat pulse and the temperature change is measured on the back face (see Figure 2).

The record shall be started before the pulse in order to determine the baseline. Care should be taken to avoid possible baseline shifts caused by the pulse.

The energy level of the heat pulse source shall produce a rise in temperature not exceeding 5 K on the back face of the test piece. If this is not the case, a new test shall be realized with a lower energy until this condition is met.

The following circumstances invalidate a test:

— failure to specify and record test conditions;

— temperature rise of the back face higher than 5 K.

9 Results

9.1 Determination of the thermal diffusivity

The value of the thermal diffusivity is determined by comparing the experimental thermogram (see Figure 1) with a set of calculated thermograms obtained by the application of a thermal model.

A number of simplifying approximations lies at the basis of the considered thermal model. These approximations impose some limitations on the range of applicability of the model and consequently some boundary conditions on the validity of the experiment (see, for example, Annex A).

NOTE In the case of a test out of symmetry axes, the evaluation of the obtained back face temperature rise is also possible in accordance with other thermal models which are derived from the Fourier equation of heat transfer (see Bibliography).

9.2 Sources of uncertainties

The relative expanded uncertainty for thermal diffusivity measurements can be calculated by taking into account different sources of uncertainties:

— physical characteristics of the specimen, such as parallelism and flatness of the faces, isotropy, homogeneity and opacity of the material;

— physical quantities, such as thickness and temperature of the specimen, time, voltage delivery and linearity of the IR detector;

— boundary conditions of the theoretical model, such as finite pulse time, laser uniformity and thermal losses.

NOTE For the temperature range 100 K to 1 673 K, the commonly relative expanded uncertainty lies between 3 % and 6 % for bulk homogeneous opaque materials by using the partial time moments method detailed in Annex A. This relative uncertainty can be strongly reduced by using an indirect statistical approach for the most important uncertainty sources, such as a Bayesian framework.

10 Test report

The test report shall contain at least the following information:

a) name of the testing establishment;

b) date of the test, unique identification of the report and of each page, the customer’s and signatory's name and address;

c) reference to this document, i.e. “Determined in accordance with ISO 19629.”;

d) description of the test specimen: material type, manufacturing code, batch number;

e) description of the equipment used;

f) direction of measurement;

g) calibration procedures if applicable;

h) methods of manufacturing of the test specimens from supplied material (if appropriate), test specimen thickness, type and thickness of coatings;

i) transient detector employed;

j) environmental conditions, i.e. vacuum, inert gas, etc.;

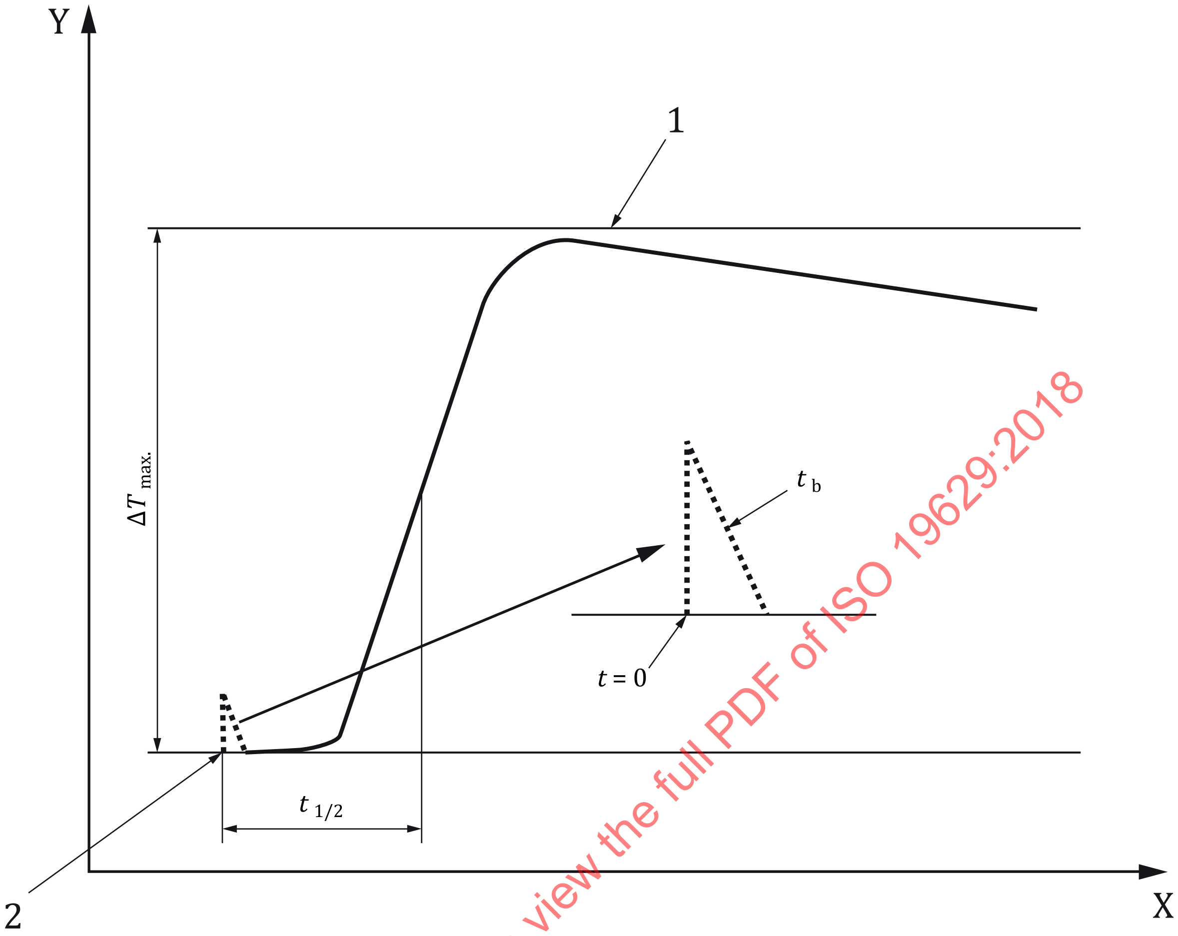
k) statement regarding the thermal expansion of the test specimen and whether or not a correction to the thickness was applied;

l) thermal model used;

m) individual values and average value of the thermal diffusivity;

n) number of tests carried out and the number of valid results obtained;

o) comments on the test or the test results.



Key

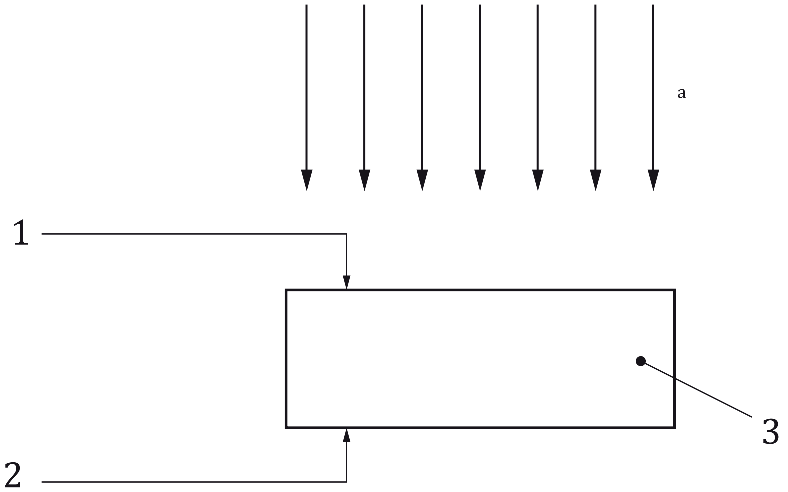
X time

Y temperature

1 maximum temperature under adiabatic conditions

2 pulse triggering $t = 0$

Figure 1 — Schematic representation of the temperature rise of the back face of the test specimen



Key

1 front face

2 back face

3 test specimen

a Energy from pulse source.

Figure 2 — Test configuration

Annex A
(informative)

Unidimensional thermal model

The model makes use of two parameters, the characteristic time and Biot number. Both parameters depend on the size and thermal properties of the test specimen.

The characteristic time is defined as h^2/a, where h is the thickness of the test specimen and a its thermal diffusivity (to be determined). The Biot number, Bi, corresponding to the test piece is defined by Formula (A.1).

$$Bi = k \cdot h/\lambda \tag{A.1}$$

where

k is the heat exchange coefficient between the test specimen and its surrounding in watts per square metre and per Kelvin, ($\mathrm{W \cdot m^{-2} \cdot K^{-1}}$);

λ is the thermal conductivity in watts per metre and per Kelvin, ($\mathrm{W \cdot m^{-1} \cdot K^{-1}}$).

$$\frac{\Delta T}{Q/\rho Ch} = 2\sum_{n=1}^{\infty} \frac{\mu_\mathrm{n}^2\left(\cos\mu_\mathrm{n} + \frac{Bi}{\mu_\mathrm{n}}\sin\mu_\mathrm{n}\right)}{\mu_\mathrm{n}^2 + 2Bi + Bi^2}\exp\left(-\mu_\mathrm{n}^2\frac{at}{h^2}\right) \tag{A.2}$$

where

ΔT is the temperature rise, in Kelvins (K);

Q is the energy absorbed per surface unit, in joules per square metre ($\mathrm{J \cdot m^{-2}}$);

T is the time, expressed in seconds (s);

ρ is the bulk density in kilograms per cubic metre ($\mathrm{kg \cdot m^{-3}}$);

C is the specific heat capacity in Joules per kilogram and per Kelvin ($\mathrm{J \cdot kg^{-1} \cdot K^{-1}}$).

The coefficients μ_n appearing in Formula (A.2) are the roots of the transcendental equation in Formula (A.3).

$$2\mu_\mathrm{n} Bi = (\mu_\mathrm{n}{}^2 - Bi^2) tg \mu_\mathrm{n} \tag{A.3}$$

This transcendental equation results from the solution of a set of differential equations that describe the heat transfer according to the unidimensional model. The roots μ_n are the eigenvalues and each of the terms in Formula (A.2) is an eigen function of the thermal problem.

From Formula (A.3), it can be observed that the values of μ_n depend on the value of the Biot number Bi. Also, the argument of the exponential factor in the series formula contains the inverse of the characteristic time. It thus appears that the theoretical thermogram depends on both the Biot number and the characteristic time. Consequently, by varying the values of both parameters, two families of thermograms can be obtained. Comparison of the theoretical curve that best fits the experimental thermogram allows determination of the numerical values of the characteristic time and of the Biot number of the test specimen. From the value of the characteristic time, the value of the thermal diffusivity is obtained directly.

A number of methods exist that allow the identification of the values of the two parameters that best fit the experimental thermogram. It is recommended that the agreement obtained by these methods is checked by plotting the reduced error (the reduced error should be properly defined here) between the experimental and the best fit theoretical thermogram as a function of time. The time dependence of the reduced error should be random. Residuals with non-random time dependence shall be rejected in the evaluation of the thermal diffusivity.

A suggested identification method is the so-called "temporal moment method". This method makes use of two moments of the thermogram, M_{-1} and M_0. These moments are defined in Formula A.4 and Formula A.5.

$$M_0 = \int_{t_{0,1}}^{t_{0,8}} \Delta T(t) dt \tag{A.4}$$

$$M_{-1} = \int_{t_{0,1}}^{t_{0,8}} \frac{\Delta T(t)}{t} dt \tag{A.5}$$

where

$\Delta T(t)$ is obtained from the experimental thermogram;

$t_{0,1}$ and $t_{0,8}$ represent the times needed to reach 0,1 $\Delta T_{\max}$ and 0,8 $\Delta T_{\max}$, respectively.

The characteristic time h^2/a according to this method is approximated by the ratio $M_0/F(M_{-1})$, where the identification function F is defined in Formula (A.6).

$$F = 0{,}085\,48 - 0{,}326\,01\,(0{,}548\,6 - M_{-1}) + 0{,}295\,92\,(0{,}548\,6 - M_{-1})^{2{,}160\,7} \tag{A.6}$$