BRITISH STANDARD

BS EN
1159-2:2003

Advanced technical ceramics — Ceramic composites — Thermophysical properties —

Part 2: Determination of thermal diffusivity

The European Standard EN 1159-2:2003 has the status of a British Standard

ICS 81.060.30

National foreword

This British Standard is the official English language version of EN 1159-2:2003. It supersedes DD ENV 1159-2:1994 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee RPI/13, Advanced technical ceramics, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

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This British Standard, was published under the authority of the Standards Policy and Strategy Committee on 5 November 2003

Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 13 and a back cover.

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Amendments issued since publication

Amd. No.	Date	Comments

ISBN 0 580 42891 5

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 1159-2

September 2003

ICS 81.060.30

Supersedes ENV 1159-2:1993

English version

Advanced technical ceramics - Ceramic composites - Thermophysical properties - Part 2: Determination of thermal diffusivity

Céramiques techniques avancées - Céramiques composites - Propriétés thermophysiques - Partie 2: Détermination de la diffusivité thermique

Hochleistungskeramik - Keramische Verbundwerkstoffe - Thermophysikalische Eigenschaften - Teil 2: Bestimmung der Temperaturleitfähigkeit

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Management Centre: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN 1159-2:2003 E

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Foreword

This document (EN 1159-2:2003) has been prepared by Technical Committee CEN/TC 184 “Advanced technical ceramics”, the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by March 2004, and conflicting national standards shall be withdrawn at the latest by March 2004.

EN 1159 consists of four parts :

— *Part 1 : Determination of thermal expansion*

— *Part 2 : Determination of thermal diffusivity*

— *Part 3: Determination of specific heat capacity*

— *Part 4: Determination of thermal conductivity*

Annex A is informative.

The document supersedes ENV 1159-2:1993.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This part of EN 1159 describes the laser flash method for the determination of thermal diffusivity of ceramic matrix composites with continuous fibre reinforcement.

The experimental conditions are such that the material behaves in an homogeneous manner for each of its axes of anisotropy and that the heat transfer occurs only by thermal conduction.

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

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

ENV 843-5, *Advanced technical ceramics — Monolithic ceramics — Mechanical tests at room temperature — Part 5 : Statistical analysis.*

ENV 13233, *Advanced technical ceramics — Ceramic composites — Notations and symbols.*

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

ISO 3611, *Micrometer callipers for external measurement.*

3 Terms and definitions

For the purposes of this European Standard, the terms and definitions given in ENV 13233 and the following apply.

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 test piece is exposed to a uniformly distributed energy pulse that is of very short duration compared to 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) 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 that 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.

NOTE 1 Measurement under vacuum will reduce convection losses.

NOTE 2 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.

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 an infrared 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.

It shall have a response time :

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

where

t_d is the response time, in second, (s)

h is the thickness, in metre, (m)

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

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) Hg/Cd/Te cell, liquid nitrogen cooled, for test specimen temperatures within the range 300 K to 800 K ;

b) PbS cell 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.

Thermocouples, when used, 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.

NOTE 1 In order to minimize heat losses, the use of the thermocouples with wires of the smallest possible diameter is recommended.

NOTE 2 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 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 8 mm and 25 mm is used.

The thickness of the specimen shall be sufficient in order to avoid influence of material homogeneity. This shall be ensured by performing tests on two series of test specimens with a thickness ratio of about 2. Recommended starting thicknesses are between 1 mm and 10 mm. Homogeneous material behaviour can be assumed when the mean values of the thermal diffusivity determined from each series do not differ by more than 10 %.

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 shall be flat and parallel. The plan parallelism of the two faces shall be better than 0,05 mm.

If the test specimen is transparent to the infrared radiation at the considered wavelength of the laser, a coating is necessary. This coating shall be opaque, absorbent, adherent and compatible with the test specimen.

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.

If a statistical evaluation is required, the number of test specimens shall be in accordance with ENV 843-5.

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 photographic paper (Polaroid type).

NOTE There is no recognised 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 0,003 $\frac{h^2}{a}$ 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 a case 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 as the front face shall be 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 test piece with the sample holder shall be as small as possible.

After the test specimen has reached 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 base line 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 it is not the case, a new test shall be realised 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

The value of the thermal diffusivity is determined by comparing the experimental thermogram (see Figure 1) to 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 to the range of applicability of the model and consequently some boundary conditions on the validity of the experiment (See for example annex A).

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. For every thermal model which is used, the range of application shall be considered. This range of application depends on the approximation which is made within the solution of the Fourier equation (see Bibliography).

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 European Standard, i.e. "Determined in accordance with EN 1159-2" ;

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

e) description of the equipment used;

f) calibration procedures if applicable;

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

h) transient detector employed;

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

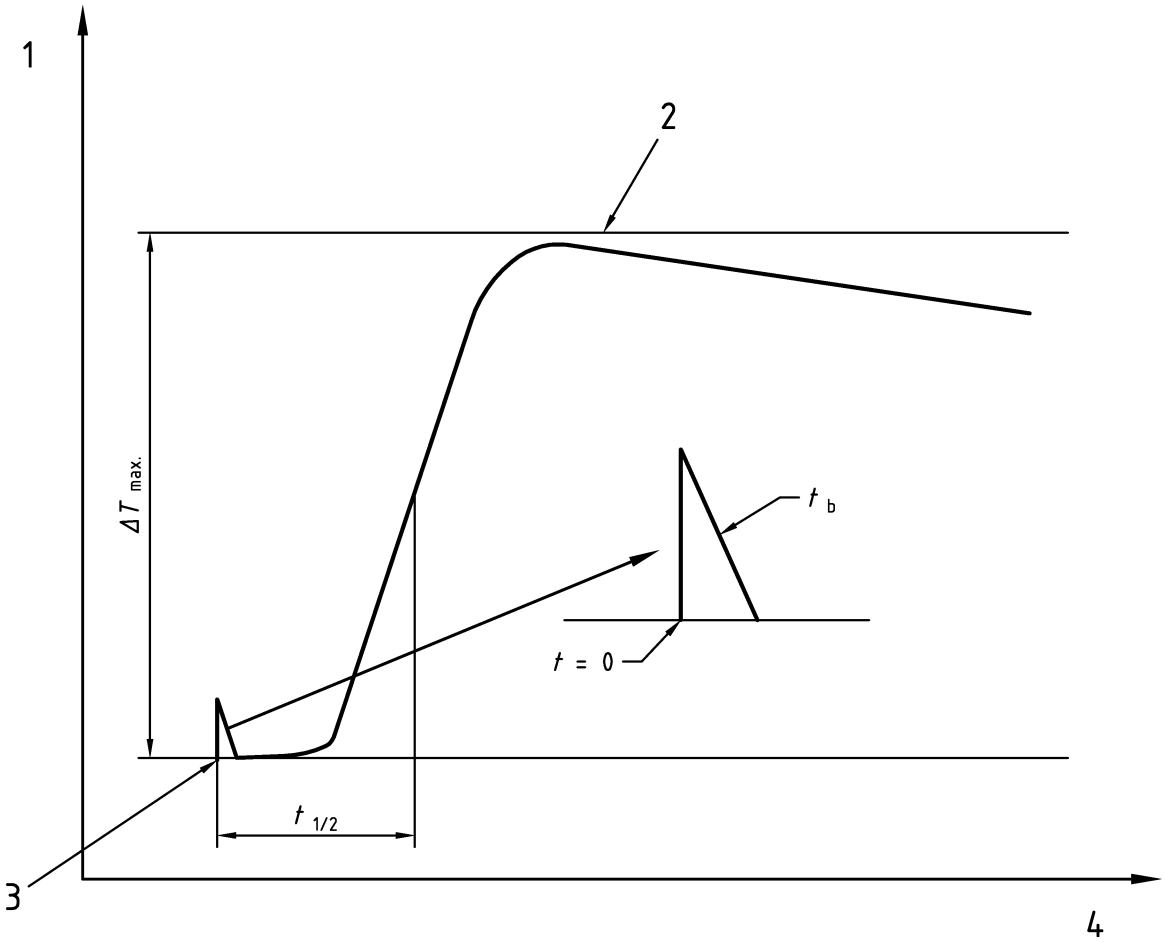
j) statement regarding the thermal expansion of the test specimen and whether or not a correction to the thickness was applied;

k) thermal model used;

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

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

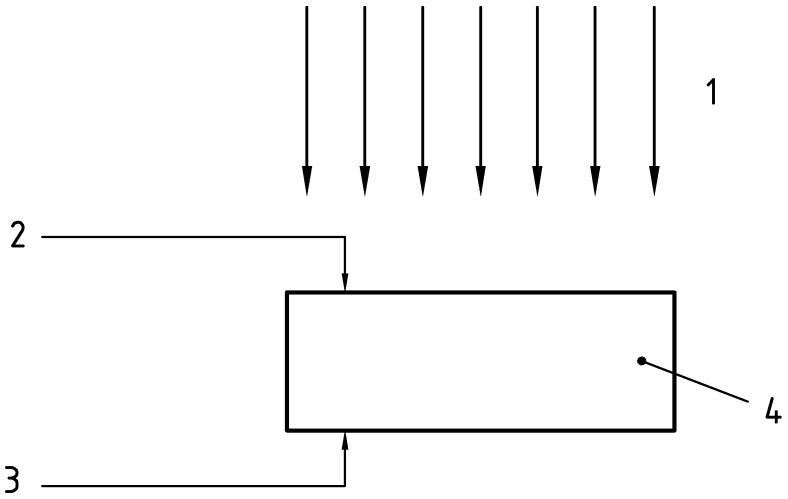
n) comments on the test or the test results.



Key

1 Temperature
2 No heat losses
3 Pulse triggering t = 0
4 Time

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



Key

1 Energy from pulse source
2 Front face
3 Back face
4 Test specimen

Figure 2 — Test configuration

Annex A
(informative)

Uni-dimensional 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 corresponding to the test piece is defined by:

$$B_i = k \cdot h / \lambda$$

where

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

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

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

where

ΔT is the temperature rise, in Kelvin, (K)

Q is the energy absorbed per surface unit, in joule by square metre, ($J.m^{-2}$)

T is the time, expressed in second, (s)

ρ is the bulk density in kilogram per cubic metre, ($kg.m^{-3}$)

C is the specific heat capacity in Joule per kilogram and per kelvin, ($J.kg^{-1}.K^{-1}$)

The coefficients μ_n appearing in equation (1) are the roots of the transcendental equation:

$$2\mu_n B_i = (\mu_n^2 - B_i^2)\ tg\ \mu_n$$

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 equation (1) is an eigen function of the thermal problem.

From the above equation, it can be observed that the values of μ_n depend on the value of the Biot number B_i. Also, the argument of the exponential factor in the series equation 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 to determine the numerical values of the characteristic time and of the Biot's 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 as follows:

$$M_0 = \int_{t_{0,1}}^{t_{0,8}} \Delta T(t)\,dt \qquad M_{-1} = \int_{t_{0,1}}^{t_{0,8}} \frac{\Delta T(t)}{t}\,dt$$

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 ΔT_{max} and 0,8 ΔT_{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 as follows:

$$F = 0{,}08548 - 0{,}32601\,(0{,}5486 - M_{-1}) + 0{,}29592\,(0{,}5486 - M_{-1})^{2{,}1607}$$

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