

# **SLOVENSKI STANDARD**

## **SIST EN 1159-2:2004**

**01-januar-2004**

**BUXca Yý U**  
**SIST ENV 1159-2:2000**

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

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

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

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

**Ta slovenski standard je istoveten z: EN 1159-2:2003**

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**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

This European Standard was approved by CEN on 1 August 2003.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and United Kingdom.

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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.

## EN 1159-2:2003 (E)

## 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  $10^{-4} \text{ m}^2 \text{ s}^{-1}$  and  $10^{-7} \text{ m}^2 \text{ s}^{-1}$ .

## 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.*

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## 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,  $\alpha$**

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 ( $\Delta T$ ) on the opposite face (back face) or a quantity directly proportional to  $\Delta T$  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 ( $\leq 5$  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  $\Delta T$  or a quantity directly proportional to  $\Delta T$  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.

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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 ( $\text{m}^2 \cdot \text{s}^{-1}$ )

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:  $\text{Bi}_2\text{Te}_3$  from 90 K to 400 K and  $\text{FeSi}_2$  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.