SLOVENSKI STANDARD
SIST-TS CEN/TS 15866:2009

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Sodobna tehniļ na keramika - Keramiļ ni kompoziti - Doloļ anje toplotne difuzivnosti keramiļ nih vlaken

Advanced technical ceramics - Ceramic composites - Determination of the thermal diffusivity of ceramic fibres

Hochleistungskeramik - Keramische Verbundwerkstoffe - Bestimmung der Temperaturleitfähigkeit von keramischen Fasern

Céramiques techniques avancées - Céramiques composites - Détermination de la diffusion thermique des fibres céramiques

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English Version

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Céramiques techniques avancées - Céramiques composites - Détermination de la diffusion thermique des fibres céramiques

Hochleistungskeramik - Keramische Verbundwerkstoffe - Bestimmung der Temperaturleitfähigkeit von keramischen Fasern

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Foreword

This document (CEN/TS 15866:2009) has been prepared by Technical Committee CEN/TC 184 “Advanced technical ceramics”, the secretariat of which is held by BSI.

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1 Scope

This Technical Specification specifies the conditions for the determination of the thermal diffusivity of single filaments of ceramic fibres parallel to the fibre axis.

This Technical Specification applies to continuous ceramic filaments taken from tows, yarns, braids and knittings.

The experimental conditions are such that the material behaves in a homogeneous manner 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 600 K. It is suitable for the measurement of thermal diffusivity values in the range between 10^{-4} $m^2 \cdot s^{-1}$ and 10^{-7} $m^2 \cdot s^{-1}$.

2 Normative references

The following referenced documents are indispensable for the application 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.

EN 843-5:2006, *Advanced technical ceramics — Mechanical properties of monolithic ceramics at room temperature — Part 5: Statistical analysis*

CEN/TR 13233:2007, *Advanced technical ceramics — Notations and symbols*

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

EN ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:2005)*

ISO 3611, *Micrometer callipers for external measurement*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in CEN/TR 13233:2007 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
$t_{½}$
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 specimen 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 uni-dimensional 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 specimen.

5.2 Test chamber

The test chamber shall be 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) 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 contain a working area in which the spatial temperature gradient is sufficiently low (≤ 5 K) to ensure a consistent temperature across the sample. In addition, it 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 reduces 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 or 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 calculated as follows:

$$t_d \leq 0{,}002\ \frac{h^2}{a} \qquad (1)$$

where

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

h is the thickness, in metre (m);

a is the thermal diffusivity, in square metre 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.

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

NOTE 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.

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 those described in Annex B). Generally, a disc-shape test specimen with 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 thickness 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 made from fibres shall be cut with their longitudinal axis coinciding with the fibre axis. A suggested method to prepare such test specimens is described in Annex B. It essentially consists of introducing fibres with a needle in a thermoplastic tube (as for example those used to protect electric cables). Once the required volume of fibres is reached, the plastic tube is heated and then shrinks, thereby exerting a sufficient pressure on the packed fibres to obtain a good degree of compaction as shown in the figures of Annex B. Disks can be cut to the desired thickness.

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 infra red 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 at each test temperature.

If a statistical evaluation is required, the number of test specimens shall be in accordance with EN 843-5:2006.

8 Procedure

8.1 Calibration of apparatus

Although 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 recognized standard reference material for thermal diffusivity measurements, although several materials are used (for example POCO graphite, ARMCO iron).

8.2 Test procedure

8.2.1 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 met, a correction of the thermogram is possible by placing the time origin at the energetic barycentre t_b of pulse (see Figure 1).