SLOVENSKI STANDARD

SIST ENV 1159-2:2000

01-december-2000

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

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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: ENV 1159-2:1993

ICS:

81.060.30	Sodobna keramika	Advanced ceramics

SIST ENV 1159-2:2000 en

EUROPEAN PRESTANDARD

PRÉNORME EUROPÉENNE

EUROPÄISCHE VORNORM

ENV 1159-2

August 1993

UDC 666.5/.6:620.1:536.21

Descriptors: Composite materials, reinforcing materials, ceramics, thermodynamic properties, détermination, diffusion, thermal conductivity

English version

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

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Ref. No. ENV 1159-2:1993 E

Contents list

Annexes

Foreword

This European prestandard has been prepared by CEN/TC 184 "Advanced technical ceramics" the secretariat of which is held by BSI.

ENV 1159 consists of three parts:

Part 1: Determination of thermal expansion
Part 2: Determination of thermal diffusivity
Part 3: Determination of specific heat capacity

CEN/TC 184 approved this European Prestandard by resolution 2-92 during its sixth meeting held in Alkmaar, Netherlands on 92-09-30.

According to the CEN/CENELEC Internal Regulations, the following countries are bound to announce this European prestandard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, United Kingdom.

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

1 Scope

This Part of ENV 1159 describes the laser flash method for the determination of thermal diffusivity of ceramic matrix composites with continuous reinforcements (1D, 2D, 3D).

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 2800 K. It is suitable for the measurement of thermal diffusivity values in the range between 10^{-4} and 10^{-7} m^2/s.

2 Normative references

This European prestandard 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 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.

EN 45001 General criteria for the operation of testing laboratories

HD 446.1S1 Thermocouples Part 1 : Reference tables

ISO 31-4 Quantities and units - Part 4: Heat

ISO 6906 Vernier callipers reading to 0,02 mm

3 Definitions, symbols, units

For the purposes of this European prestandard the following definitions apply:

3.1 Thermal conductivity: Ratio of the density of heat flow rate to the temperature gradient.

3.2 Specific heat capacity: Quantity of heat that is necessary to increase the temperature of a mass unit of a material by 1 K.

3.3 Bulk density: The ratio of the mass of the dry material of a porous body to its bulk volume.

3.4 Thermal diffusivity: Ratio of the thermal conductivity to the product of the bulk density and the specific heat capacity.

3.5 Transient half time: Time from the initiation of the pulse until the increase in the back face temperature of the test piece reaches one half of the maximum temperature increase.

3.6 Thickness: 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.

NOTE: This is most easily obtained with a flash tube.

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.

5.2 Environmental control chamber

The environmental control 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 furnace shall be either fitted with a window, transparent to the pulse radiation, or else the heat pulse source may be placed inside the furnace.

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 monitored either by a thermocouple (in accordance with HD 446-1) or by 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:

$$t_d \leq 0{,}002 \frac{e^2}{\alpha}$$

where

t_d = response time in seconds
e = thickness in metres
α = thermal diffusivity, in m^2/s

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

The infrared detector, where 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 piece temperatures within the range 300 to 800 K.

b) In/Sb cell, liquid nitrogen cooled, for test piece temperatures down to 300 K.

c) PbS cell for test piece temperatures above 500 K.

Care shall be taken that the signal comes only from the central area of the back face.

Thermocouples, where 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 HD 446.1. Electrically non-conductive material shall be coated 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 precision wire of the smallest possible diameter is recommended.

NOTE 2: The thermocouple type most often used is chromel-alumel for measurements from ambient temperature up to 1100 K. Semi-conducting couples may also be used: Bi_2-Te_3 from 90 K to 400 K, and Fe Si_2 for temperatures up to 1100 K. For temperatures over 1100 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 an accurate means of recording the temperature change versus time (before, during and after the pulse) and the time origin.

6 Test pieces

6.1 Dimensions

The size of test pieces shall be fixed to meet the requirements for application of the thermal model described in annex A. Generally a disc of a diameter between 8 mm and 25 mm is used.

The response of the specimen shall be similar to that of a homogeneous solid. This shall be ensured by performing tests on two series of test pieces with a thickness ratio of about 2. Recommended starting dimensions are between 1 mm and 10 mm thickness. 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 %.

NOTE: In order to obtain a reliable estimate of the mean value, it is recommended to test 5 test pieces per series as a minimum.