SLOVENSKI STANDARD
SIST EN 821-2:2000

01-december-2000

Advanced technical ceramics - Monolithic ceramics - Thermo-physical properties - Part 2: Determination of thermal diffusivity by the laser flash (or heat pulse) method

Advanced technical ceramics - Monolithic ceramics - Thermo-physical properties - Part 2: Determination of thermal diffusivity by the laser flash (or heat pulse) method

Hochleistungskeramik - Monolithische Keramik - Thermophysikalische Eigenschaften - Teil 2: Messung der Temperaturleitfähigkeit mit dem Laserflash- (oder Wärmeimpuls-) Verfahren

Céramiques techniques avancées - Céramiques monolithiques - Propriétés thermo-physiques - Partie 2: Détermination de la diffusion thermique par la méthode Flash laser (ou impulsion de chaleur)

Ta slovenski standard je istoveten z: **EN 821-2:1997**

ICS:

81.060.30	Sodobna keramika	Advanced ceramics

SIST EN 821-2:2000 **en**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 821-2

June 1997

ICS 81.060.99

Descriptors: ceramics, powdery materials, thermodynamic properties, tests, determination, diffusion, thermal conductivity

English version

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Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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CEN

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

Central Secretariat: rue de Stassart,36 B-1050 Brussels

Ref. No. EN 821-2:1997 E

Contents

Foreword

This European Standard 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of an identical text or by endorsement, at the latest by December 1997, and conflicting national standards shall be withdrawn at the latest by December 1997.

EN 821 consists of three Parts:

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

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, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This Part of EN 821 specifies a method for the determination of thermal diffusivity of advanced monolithic technical ceramics, to an accuracy of approximately ± 5 %. It is suitable for the measurement of thermal diffusivity values in the range 0,1 mm^2/s to 1000 mm^2/s at temperatures greater than - 180 °C.

Annex A gives the mathematical derivation of the calculations, and Annex B contains instruction on actions necessary when the calculations cannot be made in the usual way.

NOTE 1 : It is not advisable to exceed the temperature at which the test piece was manufactured.

NOTE 2 : This method involves the use of a high powered pulsed laser system or high energy photoflash equipment as well as high vacuum and high temperature furnace capability. Such equipment therefore should be operated within established safety procedures. See EN 60825.

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

EN 45001	General criteria for the operation of testing laboratories
EN 60584-1	Thermocouples Part 1 : Reference tables
EN 60584-2	Thermocouples Part 2 : Tolerances

3 Definitions

For the purposes of this Part of EN 821, the following definitions apply:

3.1 thermal diffusivity: Thermal conductivity divided by heat capacity per unit volume.

3.2 thermal conductivity: Density of heat flow rate divided by temperature gradient under steady state conditions.

3.3 specific heat: The heat capacity per unit mass.

3.4 transient half time: The time required for the temperature to rise to half of its peak or maximum.

4 Principle

Thermal diffusivity is a measure of the heat flow in a material under non-steady state conditions. It can also be related to thermal conductivity via the specific heat of the material using the relationship:

$$a = \frac{\lambda}{\rho \cdot c_p} \qquad (1)$$

where

a	thermal diffusivity in	m^2/s
λ	thermal conductivity in	$Wm^{-1}K^{-1}$
ρ	density in	kg/m^3
c_p	specific heat in	$J/(kg{\cdot}K)$

Thermal diffusivity is measured by applying a high intensity short duration heat pulse to one face of a parallel sided homogeneous test piece, monitoring the temperature rise at the opposite face as a function of time, and determining the transient half time ($t_{0,5}$). The transient temperature rise (see Annex A) is shown schematically in Figure 1. The signal from the temperature detector is recorded with an appropriate data acquisition system.

The experimental data are subject to both systematic and random errors e.g. those associated with

a) test piece thickness determination

b) time measurement on transient curve

c) response time of detectors

d) response time of recording and analysis equipment

e) trigger delays

f) non-uniform heating of the test piece

NOTE : Improvement in the accuracy can be obtained by increasing the sophistication of the data collection and analysis systems.

5 Apparatus

NOTE 1 : The essential features of the apparatus are shown in Figure 2.

5.1 Heat pulse source

The heat pulse source may be a pulsed laser, a flash tube or an electron beam. The pulse energy shall be uniform over the face of the test piece.

NOTE 2 : This is reasonably simple to achieve in the case of the flash lamp which should be housed in a totally reflecting box with a hole, and a light guide of approximately 25 mm diameter abutting the sample.

NOTE 3 : Significant errors in derived data can arise if the temperature rise exceeds 5 K, especially in materials where the thermal diffusivity is strongly temperature dependent.

The pulse source shall produce a rise in temperature not exceeding 10 K (preferably not exceeding 5 K) on the rear face of the test piece.

For measurement at high temperature, the use of a laser is recommended; flash tubes are usually restricted to a maximum of 400 °C.

NOTE 4 : Where a laser is used, it is recommended that a neodymium-glass laser system is utilized because of its excellent beam uniformity over the whole diameter. 'Footprint' paper or photographic film can be used to monitor this uniformity and also to align the beam centrally on the sample front face.

5.2 Environmental control chamber

5.2.1 General. The environmental control chamber shall be either a furnace (see 5.2.2), a cryostat (see 5.2.3), or a draught-proof enclosure (for ambient temperature measurements).

5.2.2 Furnace, capable of operation within the temperature range required, and of sufficient size to contain the specimen holder (see 5.6).

The heating elements for the furnace may be constructed from either:

a) Nickel-chrome alloy, for temperatures up to 1000 °C; or
b) Platinum or silicon carbide, for temperatures up to 1500 °C; or
c) Graphite, tantalum or tungsten, for temperatures above 1500 °C.

In steady state conditions the drift in temperature shall be less than 0,01 K/s. The temperature of the test piece shall be monitored either by a thermocouple in accordance with
EN 60584-1or by an optical pyrometer (preferably two-colour).

An appropriate inert atmosphere or vacuum shall be used when necessary to protect furnace parts and test piece holder (see 5.6) from oxidation, and to protect the test piece and its coating (see 6.3) from structure/phase changes, stoichiometric changes and compatibility problems.

NOTE 1 : Care should be taken to avoid decomposition of materials at high temperatures and under reducing conditions. At high temperatures some types of ceramics may vaporize (e.g. nitrides and silicates) or otherwise react with the environment or the applied coating.

The furnace shall either be fitted with a window, transparent to the incident heat pulse radiation, or else the heat pulse source may be placed inside the furnace, for example at temperatures where a flash lamp may be employed. The furnace shall also be fitted with a window, transparent to the emitted thermal radiation opposite the rear face of the test piece, for measurement of temperature using a pyrometer and for transmission of the transient pulse to a remote detector.

5.2.3 Cryostat, capable of temperature control to 0,01 K.

NOTE 2 : Various liquids can be used (in a vacuum flask) to provide the low temperature environment e.g. liquid nitrogen, liquid oxygen, solid carbon dioxide - acetone mixture, iced water etc., or a slow flow of boiled and pre-heated liquid nitrogen.

5.3 Transient detector

5.3.1 General. The transient detector shall be either an infra-red detector (see 5.3.2) or a thermocouple (see 5.3.3). It shall be capable of detecting changes of < 1 % of the total rear face temperature rise of the test piece with a rapid linear time response, which shall discriminate to 1 % of the half rise time of the transient ($t_{0,5}$).

5.3.2 Infra-red detector, of type appropriate to the minimum test piece temperature required e.g. a liquid nitrogen cooled indium antimonide (InSb) cell (for test piece temperatures down to 40 °C) or a lead sulphide (PbS) cell, (for test piece temperatures down to 250 °C).

The detector shall be kept at some distance from the test piece (remote from the high temperature environment) and hence a lens shall be used to focus the radiation from the centre of the rear face on to the detector. Therefore all viewing windows and lenses shall transmit radiation in the appropriate wavelength band. The sensor shall always be protected against damage or saturation from the direct laser beam energy.

5.3.3 Thermocouple, of appropriate type for the required temperature range, manufactured in accordance with the tolerances given in EN 60584-2, allowing use of the reference tables given in EN 60584-1. The wire diameter shall be 0,15 mm.

NOTE 1 : The thermocouple may serve a secondary purpose of monitoring the test piece temperature by switching into a digital thermometer.

The wire ends of the thermocouple shall be prepared to minimize heat losses from the test piece into the wires, and are pressed against the test piece by using fine (1 mm to 2 mm diameter) twin bore alumina tube and springs.

NOTE 2 : Figure 3 shows an example of a test piece and thermocouple holder suitable for use at ambient temperature and below.

Non-conducting test pieces shall be coated on the rear face (see 6.3) in order to effect the thermocouple junction, where the wires are open ended and separated by approximately 1 mm. The extra thickness of the high conductivity coating shall not increase the transient at $t_{0,5}$ by more than 1 % and this shall be checked by calculation.

NOTE 3 : The use of a number of thermocouple junctions in differential mode may be used to increase the sensitivity of measurement of the transient.

5.4 Signal amplifiers

Signal amplifiers, including spike protections, analogue-digital converters, high temperature bias circuitry. They shall have low noise and fast response so as not to introduce errors into the transient measurements. None of the electronic components shall become saturated or the signals distorted. The integration time shall be less than 0,3 ms.

5.5 Data acquisition system

The system for acquiring and storing data may be either a computer data processor (preferred) or a storage oscilloscope. The system shall be equipped with an accurate means of recording the energy pulse to initiate the recording system, for example a triggering photocell.

NOTE : The computer data processor is able to analyze several thousands of data points from the transient, and can be programmed to drive the laser and trigger systems, collect data, analyze for heat losses etc., print out results and produce plots of thermal diffusivity against temperature. The oscilloscope is not very accurate and does require a means to photograph the trace for manual analysis.

It is important in both cases to verify the accuracy of time bases and response times.

5.6 Test piece holder

For tests at near ambient temperature or below, the test piece holder may be constructed from a material of poor thermal conductivity (e.g. a plastics material). An example is shown in figure 3. At higher temperatures where plastics materials become unsuitable, the test piece holder shall be constructed of suitably refractory materials (metals, graphite, etc) in such a manner as to minimize heat transfer between it and the test piece, e.g. by allowing the test piece to be clamped at only three points on or near its periphery. For use at temperatures below room temperature, the test piece holder shall be constructed in such a way that it can be inserted into an evacuated stainless steel vessel which can be placed inside the cryostat (see 5.2.3).

When a thermocouple is employed as the transient detector, the test piece holder shall incorporate a device which pushes the two thermocouple leads into contact with the conducting rear surface of the test piece using a spring arrangement (e.g. as shown in figure 3).

The test piece holder shall also be of such design as to minimize the amount of incident energy arriving on the sides of the test piece, either directly or scattered to the transient detector, especially when an infra-red detector is employed. When using a laser energy source, the specimen holder shall be equipped with apertures to the front and rear of the test piece, the diameters of which are not more than 0,5 mm and not less than 0,2 mm smaller than the diameter of the test pieces, such that only the front face of the test piece receives the energy pulse.

When using a flash-lamp energy source and a thermocouple as a transient detector, the use of apertures is advisable to avoid spurious detector level changes immediately after the heat pulse has been fired (see clause 8).

6 Test pieces

6.1 Sampling

Test pieces or components should be sampled according to the guidance given in ENV 1006.

Whenever possible six test pieces should be cut (see 6.2) from the same bulk material to obtain a level of the material variability. Where measurements are required over a large temperature range then the minimum number of test pieces shall be prepared in each of two thicknesses, one for high temperature and one for low temperature measurement, with an overlap of at least two measurement points (for comparison). A separate test report (see clause 10) is issued for each test piece.