SLOVENSKI STANDARD
oSIST prEN ISO 19629:2022

01-januar-2022

Fina keramika (sodobna keramika, sodobna tehnična keramika) - Termofizikalne lastnosti keramičnih kompozitov - Ugotavljanje enodimenzionalne toplotne difuzivnosti z bliskovno metodo (ISO 19629:2018)

Fine ceramics (advanced ceramics, advanced technical ceramics) - Thermophysical properties of ceramic composites - Determination of unidimensional thermal diffusivity by flash method (ISO 19629:2018)

Hochleistungskeramik - Thermophysikalische Eigenschaften keramischer Verbundwerkstoffe - Bestimmung der Temperaturleitfähigkeit (ISO 19629:2018)

Céramiques techniques - Propriétés thermophysiques des composites céramiques - Détermination de la diffusivité thermique unidimensionnelle par la méthode flash (ISO 19629:2018)

Ta slovenski standard je istoveten z: **prEN ISO 19629**

ICS:

81.060.30	Sodobna keramika	Advanced ceramics

oSIST prEN ISO 19629:2022 **en,fr,de**

INTERNATIONAL STANDARD

ISO 19629

First edition
2018-08

Fine ceramics (advanced ceramics, advanced technical ceramics) — Thermophysical properties of ceramic composites — Determination of unidimensional thermal diffusivity by flash method

Céramiques techniques — Propriétés thermophysiques des composites céramiques — Détermination de la diffusivité thermique unidimensionnelle par la méthode flash

Reference number
ISO 19629:2018(E)

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Published in Switzerland

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Foreword

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Thermophysical properties of ceramic composites — Determination of unidimensional thermal diffusivity by flash method

1 Scope

This document describes the flash method for the determination of thermal diffusivity of ceramic matrix composites with continuous fibre reinforcement.

In order to conform with the unidimensional heat transfer hypothesis, the experimental conditions are defined such that the material behaves in a homogeneous manner. This involves performing tests in one symmetry axis of the composite.

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

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

ISO 3611, *Geometrical product specifications (GPS) — Dimensional measuring equipment: Micrometers for external measurements — Design and metrological characteristics*

ISO 20507, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Vocabulary*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 20507 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

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-face test piece is exposed to a uniformly distributed energy pulse that is of very short duration compared with 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 over working area width) 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 the latter 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.

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.

NOTE Measurement under vacuum will reduce convection losses and will limit the oxidation phenomena at high temperature.

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 a quantum radiation 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.

The choice of the detector depends primarily on both the temperature range in which the measurement is to be performed and the characteristic time of the sample. The first parameter sets the spectral range for which the detection sensitivity of the detector shall be maximum; the second parameter fixes its time constant.

It shall have a response time as shown in Formula (1).

$$t_d \leq 0{,}002\ h^2/a \tag{1}$$

where

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

h is the thickness, in metres (m);

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

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) HgCdTe or PbSnTe cells, liquid nitrogen cooled, for test specimen temperatures within the range 300 K to 800 K;

b) PbS or InSb cells 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.

When used, thermocouples 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.

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 a 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 1 to 8 mm is used.

The thickness of the test specimen shall be defined in relation to the characteristics of the heat pulse source to minimize the uncertainty factors coming from the poorly controlled boundary conditions such as (i) the pulse duration, (ii) the non-uniformity of the incident flux, (iii) the heat losses and (iv) the nonlinearity (see diagram reported in Annex B).

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 perpendicular to the measurement face shall be flat and parallel. The parallelism error of the two faces shall be less than 0,05 mm.

If the test specimen is transparent to the infrared radiation at the considered wavelength of the heat source, a coating is necessary. This coating shall be opaque, absorbent, adherent and compatible with the test specimen. The thickness of the coating shall be as thin as possible in order to not influence the diffusivity measurement.

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.

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 use of a photocell or a photodiode.

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 Procedure

The pulse duration shall be less than or equal to 0,002 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 cases 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 that the front face is 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 the test piece with the sample holder shall be as small as possible.

