SLOVENSKI STANDARD

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Advanced technical ceramics - Ceramic composites - Thermophysical properties - Part 1: Determination of thermal expansion

Advanced technical ceramics - Ceramic composites - Thermophysical properties - Part 1: Determination of thermal expansion

Hochleistungskeramik - Keramische Verbundwerkstoffe - Thermophysikalische Eigenschaften - Teil 1: Bestimmung der thermischen Ausdehrung

Céramiques techniques avancées - Céramiques composites - Propriétés thermophysiques - Partie 1: Détermination de la dilatation thermique

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Advanced technical ceramics - Ceramic composites - Thermophysical properties - Part 1: Determination of thermal expansion

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This European Standard was approved by CEN on 23 May 2003.

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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.

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Foreword

This document (EN 1159-1:2003) 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of an identical text or by endorsement, at the latest by January 2004, and conflicting national standards shall be withdrawn at the latest by January 2004.

This document supersedes ENV 1159-1:1993.

EN 1159 'Advanced technical ceramics – Ceramic composites – Thermophysical properties' 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*

Annexes A and B are normative.

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.

1 Scope

This Part of EN 1159 describes methods for the determination of linear thermal expansion characteristics of ceramic matrix composite materials up to 2 300 K, and is applicable to 10, 2D and nD materials.

The method describes general principles of construction calibration and operation of the equipment.

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).

EN 821-1, *Advanced technical ceramics - Monolithic ceramics - Thermo-physical properties - Part 1 : Determination of thermal expansion.*

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 17025:1999)*

ISO 3611, *Micrometer callipers for external measurement.*

ISO 6906, *Vernier callipers reading to 0,02 mm.*

3 Terms and definitions

For the purposes of this European Standard, the following terms and definitions apply.

3.1
linear thermal expansion
positive or negative change in one dimension that occurs when a material is subjected to a change in temperature

3.2
linear thermal expansion coefficient at temperature T
derivative of the length L with respect to temperature at the temperature T, divided by the length at temperature T

$$\alpha_{\mathrm{T}} = \frac{1}{L}\left(\frac{dL}{dT}\right)T \qquad (1)$$

3.3
mean linear thermal expansion coefficient between temperatures T1 and T2
linear thermal expansion between temperatures T_1 and T_2 divided by the temperature increment T_1 to T_2 and the length at temperature T_1

$$\alpha\left(T_1,T_2\right)=\frac{L\left(T_2\right)-L\left(T_1\right)}{L\left(T_1\right)} \qquad (2)$$

3.4
representative volume element (R.V.E.)
minimum volume which is representative of the material considered

4 Principle

4.1 General

A test piece is heated and subsequently cooled, either at a specified uniform rate or using defined temperature increments. Its change of length and its temperature are measured continuously or at regular frequent intervals during the imposed temperature cycle.

One of two methods may be used to determine the linear thermal expansion coefficient, either by direct measurement or by a differential method.

4.2 Direct measurement

In this method the variation in length of the test piece is measured directly. It is necessary to know the change in dimensions of the test piece support system by previous calibration.

The test piece is placed in a specimen holder and is made to contact a displacement transducer by using a push rod made of the same material as the holder. This assembly is put in a furnace. The differential expansion between the test piece and the test piece holder is measured during the increase and the decrease in temperature.

The apparatus is shown in Figure 1.

4.3 Differential method

This method consists of measuring the changes in length between a reference piece (see 6.2) and the test piece. Its is not therefore necessary to know the change in dimensions of the test piece support system.

The apparatus is shown in Figure 2.

5 Apparatus

5.1 Construction materials

The test piece holder and the push rod shall be made from thermomechanically stable materials of the same type, which shall be chemically inert and thermally compatible with the test piece material under the environmental conditions of the test.

NOTE For temperatures above 1 400 °C, it is necessary to employ a vacuum or inert gas atmosphere, with a non-oxide material appropriate for the test environment, such as a grade of dense graphite.

5.2 Heating and cooling device

Furnace, capable of working in a controlled atmosphere when required, and of controlling the temperature of the test piece to within 1 % of its mean temperature, expressed in K.

5.3 Temperature measurement

Thermocouples, in accordance with EN 60584-1, subject to the upper temperature requirements and environmental consideration, except for tungsten-rhenium couples which may be used at higher temperatures, but are not covered by EN 60584-1 should be individually calibrated. For temperature in excess of 2 000 K, infrared detectors or any suitable device may be used.

5.4 Test piece mounting

The device used shall allow free axial movement of the test piece and of the reference piece in case of differential measurement. The mechanical environment shall minimize stresses. For vertical measurement apparatus, the test pieces shall be free standing and mechanically stable on the end-plate. For measuring apparatus which is

horizontal or inclined to the horizontal, the sideways movement or twist of the test piece shall be restricted, without any restriction of axial movement, by a suitable arrangement.

5.5 System for measuring and recording the thermal expansion

System, capable of measuring displacements to an accuracy better than 0,1 μm. The system shall allow recording of the test piece temperauture and the displacement simultaneously. The system for measuring of displacements shall be periodically calibrated in accordance with annex A for direct measurement or annex B for differential measurement.

5.6 Test piece measurement

Device for measuring the test piece dimensions, with an accuracy better than 0,05 mm (e.g. micrometer in accordance with ISO 3611 or callipers in accordance with ISO 6906).

6 Specimens

6.1 Test pieces

The dimensions of the test pieces depend on the type of apparatus used. For differential measurements the test piece and the reference piece (see 6.2) shall have the same length L_0 (see Table 1).

The test piece shall be cut in such a way that the axis of desired measurement is related to the principal fibre orientations in accordance with agreement between parties to the measurement.

The end-faces of the length of the test piece shall be plane, parallel to each other and perpendicular to the long axis.

6.2 Reference pieces

Reference materials shall be chosen so that their properties are as close as possible to the properties of the material to be tested. The reference piece shall have a volume of the same order and if possible shall have the same dimensions as the test piece (see Table 1).

For measurement at high temperature (over 2 000 K) under inert atmosphere, reference materials generally used are either tungsten or highly purified graphite. Reference materials shall be procured from a certified laboratory.

NOTE NIST in the USA is one laboratory which supplies reference materials. Highly purified 'POCO' graphite is largely used as a reference for measurement at high temperature.

6.3 Dimensions

Table 1 — Recommended test piece dimensions

Dimensions in millimeters

	Material with small R.V.E. (see 3.4) such as 1D or 2D	Material with large R.V.E. (see 3.4) such as nD	Tolerances
L_0, total length	30	70	± 0,2
B, width	7	-	-
h, thickness	Depending on material and equipment	Depending on material and equipment	Depending on material and equipment
ϕ diameter	-	30	-
Parallelism	± 0,01	± 0,01	-
NOTE 1 A test piece volume of a minimum of 5 R.V.E. is recommended (see 3.4).			
NOTE 2 The shape and dimensions of the test piece depend on the structure of reinforcement. In the case of material such as 3D, a large test piece is often necessary when the representative volume element is important.			

7 Procedure

In order to simplify calibration procedures, test pieces and reference pieces of the same length should be used. If reference and test pieces have different lengths, then it is necessary to take into account a base line shift. In this case, refer to EN 821-1.

Measure the original length of the test piece at room temperature to an accuracy better than 0,2 mm.

Make sure that the equipment has been calibrated for the type of material prior to the test, according to the procedure described in annex A for direct measurement [determination of S and $\overline{\alpha}\left(A\right)$] and in annex B for differential measurement (determination of S and δ). If the test is to be performed in an inert or vacuum environment, establish the environment before commencing heating.

Position the test piece in the equipment and proceed with heating or cooling at a rate of between 1 K/min and 5 K/mins, ensuring a fairly constant temperature gradient along and through the test piece. If the temperature is changed in steps, the hold temperature shall be maintained until the length of the test piece shows no change for a period of 5 mins.

In case of large test pieces, a stepwise temperature rise is recommended.

The mean linear thermal expansion coefficient shall be calculated as the average from the results of tests on three test pieces.

8 Calculations

8.1 Direct measurement

Calculate the change in length, Δl, of a length of the sample holder material equal to the original test piece length L_0, from the expression :

$$\Delta l = L_0 \, \overline{\alpha}_A \left(T_2 - T_1 \right) \tag{3}$$

using the calculated value of Δl, obtain the change in length of Δl of the test piece from the measured displacement by $S\Delta x$: