SLOVENSKI STANDARD
SIST EN 821-1:2000

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Advanced technical ceramics - Monolithic ceramics - Thermo-physical properties - Part 1: Determination of thermal expansion

Advanced technical ceramics - Monolithic ceramics - Thermo-physical properties - Part 1: Determination of thermal expansion

Hochleistungskeramik - Monolithische Keramik - Thermophysikalische Eigenschaften - Teil 1: Bestimmung der thermischen Längenänderung

Céramiques techniques avancées - Céramiques monolithiques - Propriétés thermo-physiques - Partie 1: Détermination de la dilatation thermique

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CEN

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Contents

Foreword

This European Standard has been prepared by the 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of an identical text or by endorsement, at the latest by July 1995, and conflicting national standards shall be withdrawn at the latest by July 1995.

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

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)

1 Scope

This Part of EN 821 describes the method for the determination of the linear thermal expansion characteristics of advanced monolithic technical ceramics up to a maximum temperature of 1500 °C (see 5.2) and to a specified level of accuracy A or B as defined in table 1.

The method describes general principles of construction, calibration and operation of suitable apparatus. Specific details, including test piece dimensions, depend on the design of the apparatus. Methods of calibration are given in annexes A and B. Thermal expansion reference data are given in annex C.

2 Normative references

This European standard incorporates by dated or undated references, 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

ENV 1006 Advanced technical ceramics - Methods of testing monolithic ceramics - Guidance on the sampling and selection of test pieces

HD 446.1 S1 Thermocouples - Part 1 : Reference tables

ISO 3611 Micrometer callipers for external measurement

ISO 6906 Vernier callipers reading to 0,02 mm

3 Definitions

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

3.1 linear thermal expansion. The proportional extension which occurs when a material is heated.

3.2 linear thermal expansion coefficient. The proportional extension which occurs when a material is heated over a temperature interval of 1 K at temperature T.

3.3 mean linear thermal expansion coefficient. The average value of the thermal expansion coefficient over a temperature range T_1 to T_2.

Table 1. Requirements for accuracy levels A and B

Test method requirement	Measurement accuracy required	
	A	B
Required accuracy of result over 100 K temperature interval	$\pm$ 0,1 x 10^{-6} K^{-1}	$\pm$ 0,5 x 10^{-6} K^{-1}
Temperature variation along test piece during test	< 2 K	< 5 K
Deviation from smooth temperature ramp or hold	< $\pm$ 1 K	< $\pm$ 2 K
Expansion measurement device sensitivity and repeatability (% of specimen length)	0,001 %	0,005 %
Sensitivity of recording of thermocouple temperatures	$\pm$ 0,1 K	$\pm$ 0,5 K

4 Principle

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 is measured continuously during the heating and cooling. The percentage expansion or contraction over the required temperature range is calculated and the results presented both as a mean linear thermal expansion coefficient over chosen temperature ranges, and as a graph of thermal expansion against temperature.

5 Apparatus

5.1 General. The apparatus shall conform to the specification given below and be capable of calibration according to the procedures given either in annex A for direct measurement or annex B for differential measurement. Any suitable proprietary apparatus may be used and suitable designs are shown in figure 1.

5.2 Construction materials. For measurements from below ambient temperature to 1000 °C, transparent fused silica or fused quartz shall be used for construction of the test piece holder. For measurements from ambient temperature to temperatures above 1000 °C, an alumina ceramic of at least 99,8 % Al_2O_3 shall be used for construction of the test piece holder.

NOTE : The use of fused silica or fused quartz at temperatures above 800 °C can lead to structural changes or crystallization, and thus to changes in the thermal expansion coefficient of the apparatus. Calibration of the apparatus (see annexes A and B) should be carried out frequently, and if there is any sign of discontinuities in the expansion curves resulting from phase transitions of cristobalite (150 °C - 250 °C) or quartz (573 °C), the apparatus should be replaced.

The push rod for transmitting the displacement of the test piece to the measuring device shall be of the same material as the test piece holder.

5.3 Test-piece holder. Some possible constructions of the test piece holder are shown in figure 2. For use with round-ended test pieces (see clause 6) the outer sleeve shall have an end-plate with a surface ground flat to within 10 μm. The normal to the surface of the end-plate shall be visually square to the measurement axis. For use with flat-ended test pieces (see clause 6) the outer sleeve shall have an end-plate which is rounded to a radius of curvature of between 1 and 20 mm. The push-rod end in contact with the test piece shall have similar shape to the test piece holder end-plate.

For test-piece holders constructed from fused silica tube or rod (see 5.3) the end-plate shall be rigidly fixed to the outer sleeve by flame fusing at a point remote from the end-plate surface. Alternatively, for outer sleeves constructed from fused silica rod, the end-plate may be prepared by machining from solid material. Examples of constructions are shown in figure 2(a).

For test piece holders constructed from alumina (see 5.2) rod, the end-plate may be prepared by machining from solid material. If constructed from alumina tube, fusion is not possible.

For vertical measuring apparatus, the outer sleeve shall be fixed to a flat plate using refractory cement outside the joint as shown in figure 2(b). For non-vertical measuring apparatus in which the sleeve end is free-standing, a closed end alumina tube shall be used, and the end-plate machined to fit inside the tube end as shown in figure 2(c). The assembly shall then be fixed in position with a suitable refractory cement, and then fired to a temperature greater than 1400 °C under an axial load.

NOTE: The firing under load minimizes the risk of movement of the assembly during use. Some movement may still occur during initial use. Thermal cycling should be continued until the net movement over a thermal cycle to the highest temperature at which the apparatus is to be used is less than 1 μm.

Alternatively, an end-plate may be shaped to fit in tangential slots in the tube. If this approach is adopted, the components shall be designed to fit together with no possibility of relative movement in use, e.g. by wedging into position.

5.4 Test-piece mounting. For vertical measuring apparatus, the test pieces shall be free-standing and mechanically stable on the end-plate (see 5.3). 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.

NOTE : This may be done, for example, by

a) using a vee-block cut to fit into the test piece holder; or

b) by using test-pieces of such dimensions that either a neat sliding fit (but see clause 6) or support on two edges is obtained; or

c) using an arrangement of supporting silica glass or alumina balls, as shown in figure 3.

Where the apparatus is designed for differential measurements, two test pieces shall be suitably mounted parallel to each other, contacting on a single end-plate.

5.5 Thermocouples. The thermocouples shall be type R, S or K, in accordance with HD 446.1. One thermocouple shall be placed with its junction in contact with the surface of the test-piece near its mid-point. Two other thermocouples shall be placed at each end of the test piece, and used in differential mode as shown in figure 4, to periodically determine the temperature distribution along the test piece.

5.6 Heating or cooling device, comprising a suitable tube furnace or cooling device designed to give a uniform temperature zone of length greater than that of the test-piece during the normal thermal cycling of the test (see clause 7). The device shall heat or cool the test piece contained in its holder (see 5.3) and in any surrounding protection tube.

The variation in temperature along the length of the test piece within the device shall be determined using differential thermocouples positioned as shown in figure 4, and shall not exceed the value given in table 1 during thermal cycling.

5.7 Temperature programmer and power control unit, for temperature control of the heating and cooling device (see 5.6), incorporating a thermocouple (see 5.5) positioned in the uniform temperature zone of the device. This apparatus shall be such that for tests at a constant rate of change of temperature, deviation from a smooth rate of change shall not exceed the value given in table 1, as determined by the thermocouple in contact with the test piece. For tests at a series of steady temperatures, variations in temperature shall not exceed the value given in table 1 of the mean temperature.

5.8 Expansion measuring device, either a micrometer, dial gauge, linear displacement transducer, or an interferometer.

The device shall have a capability of measuring displacements of the push-rod relative to the specimen holder according to the level set in table 1 (see clause 6), and individual measurements shall be repeatable to this accuracy.

Where the apparatus is designed for differential measurements, the measuring device shall directly record the differential movement of the two push rods.

NOTE 1 : The difference between separately measured push rod displacements is not acceptable as a measurement.

NOTE 2 : Ideally, the expansion measuring device should have its temperature controlled to ± 1 K, using a thermostatic device such as a water jacket operating at a temperature near to, but preferably a little above room temperature. Checks should also be made to ensure that any electronic amplifiers or recording devices used such as a transducer amplifier and recording voltmeter, have outputs that are insensitive to room temperature change. If such changes in output exceed the equivalent of a displacement of 10^{-3} mm for a 10 K change in room temperature, then the room temperature should ideally be controlled to ± 1 K.

5.9 Data recording unit, providing for simultaneous recording of test piece temperature and displacement measuring device output. The sensitivity for temperature measurements shall be set according to table 1.

NOTE : This is equivalent to 1 μV for accuracy A or 5 μV for accuracy B with type R or type S thermocouples and 4 μV for accuracy A or 20 μV for accuracy B with type K thermocouples (see 5.5).

The thermocouple (see 5.5) shall have a reference cold junction which may be the temperature of the thermostatically controlled measurement unit, a separate thermostatic unit, or electronic compensation in the recording device as appropriate.

The sensitivity for displacement measurement shall be as described in 5.8.

6 Test pieces

Materials for testing should be sampled in accordance with the guidance given in ENV 1006.

The dimensions of the test pieces are dependent on the design of the apparatus (see clause 5), and in particular the temperature homogeneity of the heating or cooling device (see 5.6).

The length of the test piece shall be similar to that of any certified reference material used to calibrate the apparatus as in annex A. For the differential type of apparatus, the length of the test piece shall be within ± 0,2 mm of the length of the piece of reference material.

NOTE 1 : When the test specimen comprises coarse-grained or heavily textured materials, care should be taken that the test specimen is fully representative of the material or component for which data are required.

The cross-section of the test piece may be any suitable shape consistent with the design of the apparatus. For materials of thermal conductivity less than 10^{-1} $W(mK)^{-1}$, one dimension shall be less than 5 mm.

NOTE 2 : This minimizes transverse temperature gradient through test pieces during heating or coding.

For use with apparatus with a flat test piece holder, end plate and push rod end (see 5.3), the ends of the test piece shall be ground to a radius of curvature between 1 mm and 20 mm, so as to ensure point contact between specimen and apparatus (see figure 5), except where a vertical measuring device is used with a free-standing test piece, where the lower end of the test piece in contact with the reference base may be ground flat, perpendicular to the test piece axis. In this case the test piece shall be mechanically stable on the reference base.

For use with apparatus with a round-ended test piece holder end plate and push rod, the test piece shall have ends ground flat and parallel (see figure 5). The test piece end surfaces shall be visually square to the measurement axis. For the differential method, the reference test-piece shall be any suitable certified material, or one selected from these given in annex C and appropriate for the temperature range over which measurements are to be made.

7 Procedure

Measure the length of the test piece at room temperature, using either vernier callipers in accordance with ISO 6906, a micrometer in accordance with ISO 3611, or other suitable measuring device, to an accuracy of either:

a) ± 0,05 mm for lengths of 10 mm or above

b) ± 0,02 mm for lengths of below 10 mm.