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Fine ceramics (advanced ceramics, advanced technical ceramics) — Thermophysical properties of ceramic composites — Determination of thermal expansion

Céramiques techniques — Propriétés thermophysiques des composites céramiques — Détermination de la dilatation thermique

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Foreword

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The committee responsible for this document is ISO/TC 206, *Fine ceramics*.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Thermophysical properties of ceramic composites — Determination of thermal expansion

1 Scope

This International Standard describes methods for the determination of linear thermal expansion characteristics of ceramic matrix composite materials up to 2 300 K, and is applicable to 1D, 2D, and nD materials.

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

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

IEC 60584-1, *Thermocouples — Part 1: Reference tables*

3 Terms and definitions

For the purposes of this document, 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

$$\alpha_T = \frac{1}{L}\left(\frac{dL}{dT}\right)$$

3.3
mean linear thermal expansion coefficient between temperatures T_1 and T_2
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

$$\alpha(T_1,T_2) = \frac{L(T_2)-L(T_1)}{L(T_1)} \times \frac{1}{(T_2-T_1)}$$

3.4
representative volume element
RVE
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 can 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 (6.2) and the test piece. It 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 IEC 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 IEC 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 temperature 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 (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.

6.3 Dimensions

See Table 1.

Table 1 — Recommended test piece dimensions

Dimensions in millimetres

	Material with small RVE (3.4) such as 1D or 2D	Material with large RVE (3.4) such as nD ($n \geq 3$)	Tolerances
L_0, total length	30	70	±0,2
B, width	7	Depending on material and equipment	—
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	—
A test piece volume of a minimum of 5 RVE (3.4) is recommended.			
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.			
For nD ($n \geq 3$) composites with large RVE, thick beam specimens or cylinders with diameter ø are recommended.			
If dimensions different from the recommended ones are used, specimen volume shall be larger than 5 RVE. Tolerances: dimensions: ±0,2 - Parallelism: ±0,05.			

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 with devices (5.6).

The accuracy shall be better than

  — 0,05 mm for length of 10 mm and above, and

  — 0,02 mm for length lower than 10 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 $\bar{\alpha}(A)$], and in Annex B for differential measurement (S and δ).

— Introduce the test piece in test system and provide a good contact between the push rod and the test piece. The force to apply on the specimen depends on the test system and material, but values lower than 1 N are generally sufficient.

— Proceed with heating or cooling at a rate of between 1 K/min and 5 K/min, ensuring a fairly constant temperature gradient along the test piece. If the temperature is changed by steps, the hold temperature shall be maintained until the length of the test piece shows no change for at least 5 min before the measure.

— Record temperature T and displacement Δx.

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.

Push rod for test should not have any reaction with the test piece and the reference.

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 Formula (1):

$$\Delta l = L_0 \, \bar{\alpha}_A \left(T_2 - T_1\right) \tag{1}$$

using the calculated value of Δl, the change in length of ΔL of the specimen is obtained from the measured displacement $S\Delta x$:

$$\Delta L = S\Delta x + \Delta l \tag{2}$$

Calculate the mean linear thermal expansion coefficient of the test piece material from Formula (3):

$$\bar{\alpha} = \frac{\Delta L}{L_0 \left(T_2 - T_1\right)} \tag{3}$$

where

$\bar{\alpha}$ is the mean linear thermal expansion coefficient of the test piece material, in K^{-1};

$\bar{\alpha}_A$ is the mean linear thermal expansion coefficient correction for the apparatus over the temperature range used. It is determined from calibration (see Annex A);

L_0 is the initial length of the test piece at room temperature, in mm;

Δx is the recorded displacement (in mm, volt, etc.) over the temperature range;

S is the measurement sensitivity of the displacement recording system;

Δl is the change in length of the sample holder material, in mm;

ΔL is the change in length of the test piece, in mm;

$T_2 - T_1$ is the temperature interval in K for which the change in length is measured.

A curve of $\frac{\Delta L}{L_0}$ versus T may be constructed.

8.2 Differential method

Calculate the mean linear thermal expansion coefficient of the test piece material from Formula (4):

$$\bar{\alpha} = \frac{S\Delta x - \delta}{L_0 \left(T_2 - T_1\right)} + \bar{\alpha}(R) \times \frac{L(R)}{L_0} \tag{4}$$

where

δ is the baseline correction over the temperature range, which is introduced because of the possibility of unequal response from the two pushrods;

$L(R)$ is the initial reference piece length at room temperature, in mm;

$\bar{\alpha}(R)$ is the mean linear thermal expansion coefficient of the reference piece, in K^{-1}.

Other terms are as in 8.1.