SLOVENSKI STANDARD
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Fina keramika (sodobna keramika, sodobna tehnična keramika) - Preskusna metoda za določanje linearnega toplotnega raztezanja monolitne keramike z uporabo metode potisne palice (ISO 17562:2016)

Fine ceramics (advanced ceramics, advanced technical ceramics) - Test method for linear thermal expansion of monolithic ceramics by push-rod technique (ISO 17562:2016)

Hochleistungskeramik - Prüfverfahren zur Bestimmung der linearen Wärmeausdehnung von monolithischer Keramik mittels Schubstangen-Technik (ISO 17562:2016)

Céramiques techniques - Détermination du coefficient de dilatation thermique linéique des céramiques monolithiques par la méthode de la tige poussoir (ISO 17562:2016)

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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for linear thermal expansion of monolithic ceramics by push-rod technique

Céramiques techniques — Détermination du coefficient de dilatation thermique linéique des céramiques monolithiques par la méthode de la tige poussoir

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

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

This second edition cancels and replaces the first edition (ISO 17562:2001), which has been technically revised.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for linear thermal expansion of monolithic ceramics by push-rod technique

1 Scope

This International Standard specifies a method for the determination of the linear thermal expansion and the linear thermal expansion coefficient of monolithic ceramics from near liquid nitrogen temperature up to a maximum temperature of 2 000 °C.

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 3611:2010, *Geometrical product specifications (GPS) — Dimensional measuring equipment: Micrometers for external measurements — Design and metrological characteristics*

IEC 13385-1, *Geometrical product specifications (GPS) — Dimensional measuring equipment — Part 1: Callipers; Design and metrological characteristics*

IEC 13385-2, *Geometrical product specifications (GPS) — Dimensional measuring equipment — Part 2: Calliper depth gauges; Design and metrological characteristics*

3 Terms and definitions

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

3.1
linear thermal expansion
between temperatures T_1 and T_2 is the ratio $\Delta L/L_0$, where $\Delta L = (L_2 - L_1)$ and L_0 = specimen length at room temperature

Note 1 to entry: When the temperature has changed from T_1 to T_2, assume that the length of specimen changes from L_1 to L_2.

3.2
mean linear thermal expansion coefficient
$\bar{\alpha}$
linear thermal expansion (3.1) divided by $\Delta T = (T_2 - T_1)$ to produce the quotient $\bar{\alpha} = \Delta L / (L_0 \cdot \Delta T)$

3.3
instantaneous linear thermal expansion coefficient
α
value of $\bar{\alpha}$ (3.2) at the limit of $T_2 \rightarrow T_1$

$$\alpha = \lim_{T_2 \rightarrow T_1} \left[\bar{\alpha}\right]$$

4 Principle

A specimen of known size is heated/cooled to a specific temperature at a controlled temperature rate in a known atmosphere under a minimal load. During the heating and cooling, the length and the temperature of the specimen are monitored. The change in dimension of the specimen across a given temperature region is used to calculate a linear thermal expansion coefficient or an instantaneous linear thermal expansion coefficient against temperature.

5 Apparatus

5.1 Micrometer callipers, in accordance with ISO 3611 or vernier callipers in accordance with IEC 13385-1 and IEC 13385-2 for measuring the specimen length, L_0, to an uncertainty of 0,1 % at 20 °C, (see ISO 3611:2010, Clause 2).

5.2 Displacement measuring device, for determining the specimen length change accompanying the temperature change having a sensitivity of $1 \times 10^{-5} \times L_0$ (see 6.1). The contact force of the push-rod to the specimen shall be adjustable. Typical values for the contact force are between 0,1 N and 1 N.

5.3 Specimen support system, to ensure that the specimen is held firmly in position by a contact force not exceeding 1 N [see Clause 7 c)], in order to maintain mechanical stability throughout measurement.

5.4 Heating or cooling device, having the capability of attaining a temperature homogeneity within ±2 °C below 1 000 °C and ±5 °C between 1 000 °C and 2 000 °C over the whole specimen length.

NOTE There is no device available that covers the full temperature range from near liquid nitrogen temperature up to a maximum temperature of 2 000 °C. It is necessary to choose the equipment according the required temperature range. Furnaces are available for different temperature ranges as from −150 °C to 1 000 °C and from room temperature to 1 500 °C or to 2 000 °C.

Liquid nitrogen is the most practical coolant for the cooling device. To realize defined heating or cooling rates, the furnace should be equipped with a cooling coil and a heating element. By means of the cooling coil, a constant cooling can be achieved and by means of the heating element, defined heating or cooling rates can be realized.

5.5 Temperature controlling device, to enable the temperature of the specimen to be controlled, upon heating or cooling to 5 °C/min or preferably lower rate or step-wise temperature changes (see [Clause 7 e)] over the whole measurement range.

5.6 Temperature measuring device, to allow the temperature of the specimen to be measured with an uncertainty of less than 2 °C within the measurement range. A thermocouple of appropriate type is usually used. Care shall be taken to ensure that the thermocouple tip is in close proximity to the specimen.

The contact force of the push-rod to the specimen shall be adjustable between 0,1 N and 1 N.

6 Specimens

6.1 Test specimen

The shape and dimension of the test specimen usually depend on the type of specimen support system. However, its shape is usually in the form of a square or circular rod. For the case of a square rod, the width and thickness shall be approximately 5 mm. If a circular rod is being used, the diameter shall be approximately 5 mm. In both cases, the length of the rod shall be at least 1×10^5 times the sensitivity of the displacement measuring device (see 5.2) calculated as at least 10 mm in the case of 0,1 µm sensitivity device. The end faces of the test specimen shall be appropriate to the design of the measurement apparatus and should either be flat, parallel and perpendicular to length or gently

rounded to provide localized contact with the test system to minimize off-axis movement. At least two test specimens should be prepared.

6.2 Reference specimen

A reference specimen is used to obtain the calibration data to correct the measured change in length of an unknown test specimen. $\bar{\alpha}$ of the reference specimen shall be known over the test temperature range. The correction to be applied to the unknown test specimen is obtained by calibration using a reference specimen.

Reference specimens are usually prepared from materials with high purity (99,99 %; crystallographically cubic and thus have isotropic thermal expansivity), pure crystalline alumina (at least 99,8 % Al_2O_3, density >3,70 g/cm^3), or fine-grained isotropic graphite as shown in Annex A. The shape and the dimensions of the reference specimen shall be similar within ±0,2 mm to those of the unknown test specimen.

Alumina is not a good reference because the thermal expansion characteristics can vary with source because of crystallographic texturing from the process method used to manufacture it. Any test-pieces used shall be independently certified. There are several varieties of fine-grained isotropic graphite available from different suppliers. So, also graphite can only be used as a reference with certification of the specific grade.

7 Procedure

7.1 General

Care should be taken to select push-rod and hold materials that will not react with the test specimen. Reference to phase diagrams or similar technical literature is advised. If there is any indication of remarkable reaction, the test results should be discarded.

7.2 Procedure for a single-rod dilatometer

a) Remove surface contamination and adherent debris from the surface of the test specimen. Using the micrometer callipers (5.1), determine the length L_0 of the test specimen to an accuracy of 0,1 % or ±0,005 mm (whichever is smaller) at room temperature, and determine room temperature to an accuracy of ±0,5 °C.

b) Remove surface contamination and adherent debris from the mounting base, and place the test specimen in the specimen holder to ensure mechanical stability.

c) Contact the push-rod gently on the end of the test specimen and apply a load of between 0,1 N and 1 N to the test specimen.

NOTE 1 It is advised to use a test load, as low as possible to avoid possible confounding errors due to test part deformation or creep at high temperatures.

d) The measurement atmosphere is air under a constant flow rate. If oxidation of the test specimen and/or of the specimen holder (as in case of the graphite specimen holder) affects measurement, use nitrogen, inert gas or vacuum.

NOTE 2 It is advised to use the minimum gas flow possible to avoid cooling of the temperature sensor and potential related measurement errors.

NOTE 3 Using nitrogen in equipment with graphite furnaces/graphite specimen holder at temperatures above 1 700 °C can result in the formation of cyanide substances. This requires caution during operation of the equipment under the mentioned conditions.

e) Change the temperature at a specified uniform rate of 5 °C/min or preferably less by means of the temperature controlling device (5.5), or by using defined step-wise temperature increments.

NOTE 4 It is advised to use the lowest practical heating rate, to avoid thermal lag between the temperature sensor and the test part. This is particularly important for materials of high density or low thermal conductivity.

f) Using the displacement measuring device (5.2) and the temperature measuring device (5.6), continuously record the whole process of the change of length of test specimen at temperature *T*.

g) The measurement shall be carried out in at least two thermal cycles without removing the test specimen.

h) All thermal expansion measurements (measurement of test specimen, measurement of reference specimen and measurement of base line variation) shall be carried out under nominally identical conditions.

7.3 Procedure for a differential type dilatometer

a) Remove surface contamination and adherent debris from the surface of the test specimen and reference specimen. Using the micrometer callipers (5.1), determine the length L_0 of the test specimen and reference specimen to an accuracy of 0,1 % or ±0,005 mm (whichever is smaller) at room temperature, and determine room temperature to an accuracy of ±0,5 °C.

b) Remove surface contamination and adherent debris from the mounting base, and place the test specimen and reference specimen in the specimen holder, to ensure mechanical stability.

c) Contact the push-rod gently on the end of the test specimen and a reference push-rod on the end of the reference specimen and apply a load of between 0,1 N and 1 N to the test specimen and reference specimen.

NOTE 1 It is advised to use a test load, as low as possible to avoid possible confounding errors due to test part deformation or creep at high temperatures.

d) The measurement atmosphere is air under a constant flow rate. If oxidation of the test specimen and/or of the specimen holder (as in case of the graphite specimen holder) affects measurement, use inert gas or a vacuum.

NOTE 2 It is advised to use a gas flow, as low as possible to avoid cooling of the temperature sensor and potential related measurement errors.

e) Change the temperature at a specified uniform rate of 5 °C/min or preferably less by means of the temperature controlling device (5.5), or by using defined step-wise temperature increments.

NOTE 3 It is advised to use the lowest practical heating rate, to avoid thermal lag between the temperature sensor and the test part. This is particularly important for materials of high density or low thermal conductivity.

f) Using the displacement measuring device (5.2) and the temperature measuring device (5.6), continuously record the whole process the differential length change between test specimen and reference specimen at temperature *T*.

g) The measurement shall be carried out in at least two thermal cycles without removing the test specimen.

h) All thermal expansion measurements (measurement of test specimen, measurement of reference specimen and measurement of base line variation) shall be carried out under nominally identical conditions.

8 Expected uncertainty level

An expected level of uncertainty is defined in Table 1.

