DRAFT INTERNATIONAL STANDARD

ISO/DIS 17562

ISO/TC **206**	Secretariat: **JISC**
Voting begins on: **2015-09-01**	Voting terminates on: **2015-12-01**

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

Céramiques techniques — Méthode d'essai pour le coefficient d'expansion thermique linéaire des céramiques monolithiques par la technique du poussoir de soupape

ICS: 81.060.30

Reference number
ISO/DIS 17562:2015(E)

COPYRIGHT PROTECTED DOCUMENT

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 17562 was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

This second/third/... edition cancels and replaces the first/second/... edition (), [clause(s) / subclause(s) / table(s) / figure(s) / annex(es)] of which [has / have] 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 2000 °C.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 3611:1978, *Micrometer callipers for external measurement*

ISO 6906:1984, *Vernier callipers reading to 0,02 mm*

ISO 7991:1987, *Glass — Determination of coefficient of mean linear thermal expansion*

ISO 17139:2014, *Fine Ceramics (advanced ceramics, advanced technical ceramics) – Thermophysical properties of ceramic composites – Determination of thermal expansion*

BS 1902-5.14, *Methods of testing – Refractory materials – Part 5. Refractory and thermal properties – Section 5.14 Determination of thermal expansion (temperatures up to 1500 °C) (methods 1902-514)*

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

IEC 60584-2:1982, *Thermocouples — Part 2: Tolerances*

3 Terms and definitions

For the purposes of this International Standard, 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 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

$\bar{a}$ between temperatures T_1 *and* T_2 is the linear thermal expansion divided by ΔT (= $T_2 - T_1$) to produce the quotient $\bar{a} = \Delta L / (L_0 \cdot \Delta T)$

3.3 instantaneous linear thermal expansion coefficient

α value of $\bar{a}$ at the limit of $T_2 \rightarrow T_1$

$$a = \lim_{T_2 \rightarrow T_1} [\bar{a}]$$

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 ISO 6906 for measuring the specimen length, L_0, to an uncertainty of 0,1 % at 20 °C, (see clause 2 of ISO 3611:1978).

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 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 1 There is no device available that covers the full temperature range from near liquid nitrogen temperature up to a maximum temperature of 2000 °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 1000 °C and from room temperature to 1500 °C or to 2000 °C.

NOTE 2 Liquid nitrogen is the most practical coolant for the cooling device. To realise defined heating or cooling rates the furnace has to 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 realised.

5.5 Temperature controlling device, to enable the temperature of the specimen to be controlled, upon heating or cooling to 5 °C/min or step-wise temperature changes (see 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 (see clause 2 of IEC 60584-22:1989). 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 localised contact with the test system to minimise off-axis movement. At least two test specimens should be prepared.

6.2 Reference specimen

Reference specimen used to obtain the calibration data to correct the measured change in length of an unknown test specimen. The linear thermal expansion coefficient 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 (POCO-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.

7 Procedure

NOTE Care should be taken to select push rod and hold materials which 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.

7.1 Procedure for a single-rod dilatometer

a) Remove surface contamination and adherent debris from the surface of the specimen. Using the micrometer callipers (5.1), determine the length L_0 of the 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 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 specimen.

NOTE 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 specimen and/or of the specimen holder (as in case of the graphite specimen holder) affects measurement, use nitrogen, inert gas or vacuum.

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

NOTE Using nitrogen in equipment with graphite furnaces/graphite specimen holder at temperatures above 1700 °C may 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 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 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.

7.2 Procedure for a differential type dilatometer

a) Remove surface contamination and adherent debris from the surface of the specimen and reference specimen. Using the micrometer callipers (5.1), determine the length L_0 of the 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 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 specimen and reference specimen.

NOTE It is advised to use a low a test load 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 specimen and/or of the specimen holder (as in case of the graphite specimen holder) affects measurement, use inert gas or a vacuum.

NOTE It is advised to use the minimum gas flow 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 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 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.

Table 1 — Uncertainty level with requirements in temperature and length measurements

Element	Required measurement uncertainty
Expected uncertainty against linear thermal expansion coefficient of 1×10^{-5} °C^{-1} over 100 °C temperature interval	2×10^{-7} °C^{-1}
Temperature determination	2 °C
Sensitivity of the measuring device (L_0: specimen length at room temperature)	$1 \times 10^{-5}\ L_0$

Reference data for thermal expansion are given in annex A. The method for calculating the thermal expansion is given in annex B. Schematics of the measuring apparatus are described in annex C.

9 Calculation of results

The linear thermal expansion and the mean linear thermal expansion coefficient between temperatures (T_1, T_2) shall be calculated from the following equations:

$$\frac{\Delta L_{sp}}{L_0} = \frac{\Delta L_{sp,m} - \Delta L_{ref,m} + \Delta L_{ref}}{L_0} \quad (1)$$

$$\bar{a} = \frac{\Delta L_{sp,m} - \Delta L_{ref,m}}{L_0 \Delta T} + \bar{a}_{ref} \quad (2)$$

where

ΔL_{sp} is the change of length of the specimen between T_1 and T_2;

L_0 is the specimen length at room temperature;

$\Delta L_{sp,m}$ is the difference of indication of displacement measuring device at T_1 and T_2 when the specimen is measured;

$\Delta L_{ref,m}$ is the difference of indication of displacement measuring device at T_1 and T_2 when the reference specimen is measured;

ΔL_{ref} is the calculated length change of the reference specimen between T_1 and T_2;

$\bar{a}$ is the mean linear thermal expansion coefficient of specimen between T_1 and T_2 (°K^{-1});

ΔT is the temperature change of specimen $T_2 - T_1$ in degrees Celsius;

$\bar{a}_{ref}$ is the calculated mean linear thermal expansion coefficient of the reference specimen between T_1 and T_2 (°K^{-1}).

The recommended values for linear thermal expansion of reference specimens, ΔL_{ref}, are shown in Table A1. Mean linear thermal expansion coefficient, $\bar{a}_{ref}$, can be calculated from ΔL_{ref}. The method used to derive equations (1) and (2) is described in annex B.