INTERNATIONAL STANDARD

ISO 17562

First edition
2001-10-15

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

Reference number
ISO 17562:2001(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

Printed in Switzerland

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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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

Annexes A and B form a normative part of this International Standard. Annex C is for information only.

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 1 500 °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*

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 ΔL = $(L_2 - L_1)$ 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
$\bar{\alpha}$
between temperatures T_1 *and* T_2 is the linear thermal expansion divided by ΔT $(= T_2 - T_1)$ to produce the quotient $\bar{\alpha} = \Delta L/(L_0 \times \Delta T)$

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

$$\alpha = \lim_{t_2 \rightarrow t_1} [\bar{\alpha}]$$

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 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 $\pm$ 2 °C below 1 000 °C and $\pm$ 5 °C between 1 000 °C and 1 500 °C over the whole specimen length.

NOTE Liquid nitrogen is the most practical coolant for the cooling device.

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. At least two test specimens should be prepared [see 7, g)].

6.2 Reference specimen

The linear thermal expansion coefficient of the reference specimen shall be known over the test temperature range. However, if such a specimen is not available it is acceptable to use materials with high purity (99,99 %) as shown in annex A. These materials are crystallographically cubic and thus have isotropic thermal expansivity. The shape and the dimensions shall be similar to those of the test specimen.

7 Procedure

The following procedure is valid for a single-rod dilatometer. Additional words in square brackets are for a differential type of dilatometer.

a) Using the micrometer callipers (5.1), determine the length L_0 of the specimen [and the reference specimen] to an accuracy of 0,1 % at 20 °C.

b) Remove surface contamination and adherent debris from the surface of the specimen [reference specimen] and mounting base, and place the specimen [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].

d) The measurement atmosphere is air under a constant flow rate. If oxidation of the specimen affects measurement, use nitrogen, inert gas or a vacuum.

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.

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 [or 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, and at least two individual test pieces shall be measured (see 6.1).

h) All thermal expansion measurements (measurement of test specimen, measurement of reference specimen, and measurement of baseline 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 20 °C)	$1 \times 10^{-5}\ L_0$

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:

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

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

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;

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

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

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

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

Calculate the average and standard deviation of measured values by plural measurement runs. If the deviation from the average is significantly greater than the standard deviation, investigate the cause of that data and repeat the measurement.

10 Calibration of apparatus

10.1 General

The measuring apparatus shall be re-calibrated periodically at regular intervals to ensure that the whole measuring system is functioning correctly. Calibrate regularly and whenever mechanical parts are changed.

10.2 Calibration of the displacement measuring device

The output of the displacement measuring device shall be calibrated by using micrometer callipers attached to the apparatus.

10.3 Calibration of the temperature measuring device

Thermocouples shall be replaced at regular intervals, or be re-certified after use at high temperatures or in corrosive environments.

11 Test report

The test report shall include at least the following:

a) details of the specimens and related data;

b) shape, dimensions and number of specimens;

c) type of measuring apparatus used;

d) measurement conditions (type of temperature change — constant heating rate or stepwise, load applied to specimen, type and flow rate of gas used for measurement atmosphere);

e) shape and dimensions of reference specimen and value of linear thermal expansion or mean linear thermal expansion coefficient used;

f) linear thermal expansion, L/L_0, mean linear thermal expansion coefficient, $\bar{\alpha}$, or instantaneous linear thermal expansion coefficient, α, over the required temperature range with their uncertainties;

g) thermal expansion curve;

h) date of measurement;

i) comments related to the measurements or the measurement results.

Annex A
(normative)

Reference data for thermal expansion

Table A.1 indicates the recommended value of linear thermal expansion of silicon (available to 700 °C in air, 1 000 °C in inert conditions), tungsten (available to 300 °C in air, 1 500 °C in inert conditions), platinum (available to 1 300 °C in air or in inert conditions) and copper (available to 300 °C in air, 800 °C in inert conditions). The data given in Table A.1 have been calculated to a level of uncertainty of ± 1 %, from referenced sources listed in the bibliography.

Table A.1 — Linear thermal expansion reference data (Unit:10^{-6})

Temperature		Material (99,99 % purity)			
°C	K	$\Delta L/L_0$ from 20 °C to temperature			
		Silicon	Tungsten	Platinum	Copper
–233	40	–217	–875		–3 235
–213	60	–223	–850		–3 158
–193	80	–232	–811		–3 018
–173	100	–240	–760		–2 829
–153	120	–244	–700		–2 605
–133	140	–242	–633		–2 353
–113	160	–232	–560		–2 080
–93	180	–214	–482		–1 792
–73	200	–190	–401		–1 492
–23	250	–101	–189		–707
0	273	–49	–88		–331
20	293	0	0	0	0
50	323	80	134	266	500
100	373	229	359	720	1 354
150	423	564	814	1 652	2 228
200	473	564	814	1 652	3 121
250	523	744	1 045	2 128	4 033
300	573	930	1 278	2 610	4 961
350	623	1 122	1 515	3 097	5 907
400	673	1 317	1 754	3 589	6 870
450	723	1 516	1 996	4 087	7 852
500	773	1 718	2 240	4 591	8 853
600	873	2 131	2 733	5 617	10 919
700	973	2 554	3 232	6 674	13 072
800	1 073	2 987	3 736	7 766	15 323
900	1 173	3 427	4 250	8 896	17 688
1 000	1 273	3 875	4 775	10 063	
1 100	1 373		5 311	11 264	
1 200	1 473		5 858	12 500	
1 300	1 573		6 415	13 777	
1 400	1 673		6 984	15 111	
1 500	1 773		7 571	16 507	
1 600	1 873		8 183		
1 700	1 973		8 803		

Annex B
(normative)

Method for deriving equations (1) and (2) for use with a single-rod type (or differential expansion type) instrument

When the temperature change $\Delta T = T_2 - T_1$ is experienced by the specimen, the indication $\Delta L_{sp,m}$ of the displacement measuring device is given by the equations:

$$\Delta L_{sp,m} = \Delta L_{sp} - \Delta L_{holder} + \Delta L_{bl} \quad \text{(B.1)}$$

for a single-rod type of instrument, or

$$\Delta L_{sp,m} = \Delta L_{sp} - \Delta L_{ref} + \Delta L_{bl} \quad \text{(B.1′)}$$

for a differential expansion type instrument,

where

ΔL_{sp} is the change of length of specimen;

ΔL_{holder} (or ΔL_{ref}) is the change of length of specimen holder (or reference specimen) due to this temperature change;

ΔL_{bl} is the base line variation.

To obtain the change of length of supporting tube and baseline variation (or the baseline variation), place the reference specimen at the position where the specimen is usually placed. Make the measurement using conditions identical to those used for measuring the test specimen. Measure the indication of the displacement measuring device against the temperature change, ΔT.

The indication $\Delta L_{ref,m}$ is expressed by the equations:

$$\Delta L_{ref,m} = \Delta L_{ref} - \Delta L_{holder} + \Delta L_{bl} \quad \text{(B.2)}$$

for a single-rod type of instrument, or

$$\Delta L_{ref,m} = \Delta L_{bl} \quad \text{(B.2′)}$$

for a differential expansion type instrument.

From the equations (B.1) and (B.2), or (B.1′) and (B.2′), the linear thermal expansion is expressed by the following equation:

$$\frac{\Delta L_{sp}}{L_0} = \frac{\Delta L_{sp,m} - \Delta L_{ref,m} + \Delta L_{ref}}{L_0} \quad \text{(B.3)}$$

If this is expressed in the form of mean linear thermal expansion coefficients, the following equation is given.

$$\bar{\alpha} = \frac{\Delta L_{sp}}{L_0 \Delta T}$$

$$= \frac{\Delta L_{sp,m} - \Delta L_{ref,m}}{L_0 \Delta T} + \bar{\alpha}_{ref} \quad \text{(B.4)}$$

where

$$\bar{\alpha}_{ref} = \frac{\Delta L_{ref}}{L_0 \Delta T} \quad \text{(B.5)}$$