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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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Contents

Page

Foreword

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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.

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 Sensitivity of the measuring device (L_0: specimen length at room temperature).	2 °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

$\Delta L_{sp}/L_0$ and $\bar{\alpha}$ between temperatures (T_1, T_2) shall be calculated from Formulae (1) and (2):

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

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

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 (K^{-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 (K^{-1}).

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

10 Calibration of apparatus

10.1 General

The measuring apparatus shall be re-calibrated periodically at regular intervals, at least annually, 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 calibrated or replaced at regular intervals, e.g. after each series of replicate tests, or be re-certified after use at high temperatures or in corrosive environments.

10.4 Measurement of base line variation

This calibration is carried out by using a test specimen made of the same material as the push-rod, or by moving the push-rod to come into contact with the end plate. A test is carried out according to the procedure given in Clause 7 and the displacement recorded is then due to different temperature distributions along the length of the push-rod and the test specimen holder.

NOTE For silica apparatus, the base line variation is usually quite small, but for alumina and graphite it can be larger because of the higher thermal conductivity and higher thermal expansion. The base line variation can be either positive or negative. The above analysis assumes that at a maximum heating rate of 5 °C/min, the base line variation is consistent and is due solely to the apparatus, not to any thermal lag in the test specimen.

11 Test report

The test report shall be in accordance with the reporting provisions of ISO/IEC 17025 and shall include at least the following:

a) a reference to this International Standard, i.e. ISO 17562;

b) details of the specimens and related data;

c) shape, dimensions and number of specimens;

d) type of measuring apparatus used;

e) 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);

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

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

h) thermal expansion curve;

i) date of measurement;

j) 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), copper (available to 300 °C in air, 800 °C in inert conditions), pure crystalline alumina (≥99,8 %), and fine-grained isotropic graphite, (available to 2 000 °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[6]	Tungsten[6]	Platinum[5]	Copper[6]	Alumina[4]	Graphite[9]
−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		−136
20	293	0	0	0	0	0	0
50	323	80	134	266	500	180	204
100	373	229	359	720	1 354	490	546
150	423	394	584	1 187	2 228	820	890
200	473	564	814	1 652	3 121	1 170	1 237
250	523	744	1 045	2 128	4 033	1 530	1 587
300	573	930	1 278	2 610	4 961	1 900	1 942
350	623	1 122	1 515	3 097	5 907	2 300	2 301
400	673	1 317	1 754	3 589	6 870	2 700	2 665
450	723	1 516	1 996	4 087	7 852	3 110	3 034
500	773	1 718	2 240	4 591	8 853	3 540	3 409
600	873	2 131	2 733	5 617	10 919	4 420	4 177
700	973	2 554	3 232	6 674	13 072	5 320	4 970
800	1 073	2 987	3 736	7 766	15 323	6 250	5 789
900	1 173	3 427	4 250	8 896	17 688	7 200	6 634
1 000	1 273	3 875	4 775	10 063		8 180	7 506
1 100	1 373		5 311	11 264		9 200	8 404
1 200	1 473		5 858	12 500		10 180	9 327

Table A.1 *(continued)*

Temperature		Material (99,99 % purity)					
°C	K	$\Delta L/L_0$ from 20 °C to temperature					
		Silicon[6]	Tungsten[6]	Platinum[5]	Copper[6]	Alumina[4]	Graphite[9]
1 300	1 573		6 415	13 777		11 120	10 277
1 400	1 673		6 984	15 111		12 110	11 250
1 500	1 773		7 571	16 507		13 050	12 249
1 600	1 873		8 183				13 271
1 700	1 973		8 803				14 318
1 800	2 073						15 390
1 900	2 173						16 488
2 000	2 273						17 614

Annex B
(normative)

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

When the temperature change $\Delta T = T_2\text{-}T_1$ is experienced by the test specimen, the indication $\Delta L_{sp,m}$ of the displacement measuring device is given by Formulae (B.1) and (B.2):

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

for a single-rod type of instrument, or

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

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 base line variation (or the base line 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 $\Delta L_{sp,m}$ is expressed by Formulae (B.3) and (B.4):

$$\Delta L_{ref,m} = \Delta L_{ref} - \Delta L_{holder} + \Delta L_{bl} \quad \text{(B.3)}$$

for a single-rod type of instrument, or

$$\Delta L_{ref,m} = \Delta L_{bl} \quad \text{(B.4)}$$

for a differential expansion type instrument.

From Formulae (B.1) and (B.3), or (B.2) and (B.4), the linear thermal expansion is expressed by Formula (5):

$$\frac{\Delta L_{\mathrm{sp}}}{L_0} = \frac{\Delta L_{\mathrm{sp,m}} - \Delta L_{\mathrm{ref,m}} + \Delta L_{\mathrm{ref}}}{L_0} \tag{B.5}$$

If this is expressed in the form of $\bar{\alpha}$, Formula (B.6) is given.

$$\bar{\alpha} = \frac{\Delta L_{\mathrm{sp}}}{L_0 \Delta T} = \frac{\Delta L_{\mathrm{sp,m}} - \Delta L_{\mathrm{ref,m}}}{L_0 \Delta T} + \bar{\alpha}_{\mathrm{ref}} \tag{B.6}$$

where

$$\bar{\alpha}_{\mathrm{ref}} = \frac{\Delta L_{\mathrm{ref}}}{L_0 \Delta T} \tag{B.7}$$

Annex B assumes that the length of the reference specimen is identical to that of the test specimen. In the situation where that cannot be achieved, it is permitted to measure the baseline displacement ΔL_{bl} directly by use of no test specimen, or by use of a test specimen of the same material as that of the push-rod.

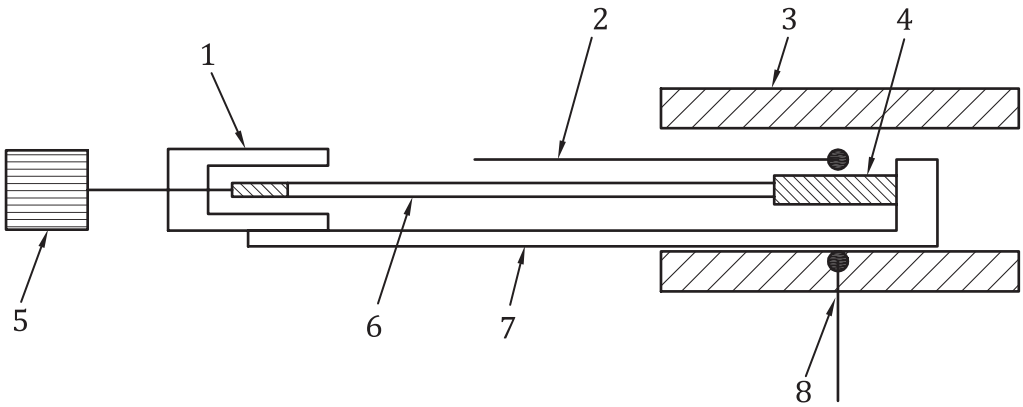
Annex C
(informative)

Suitable apparatus for the dilatometric measurement

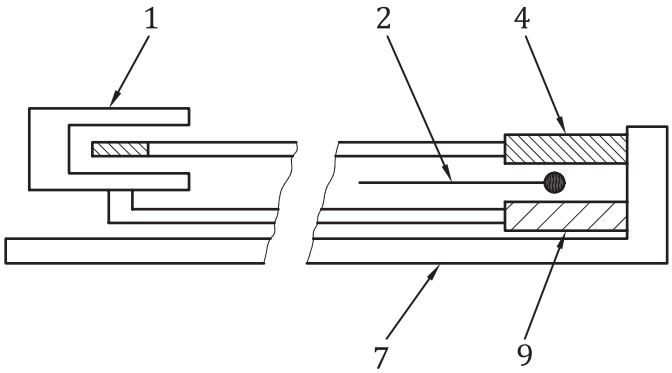
A suitable apparatus for the dilatometric measurement is shown in Figure C.1 and consists of the following.

The structure of the apparatus of a single-rod type [see Figure C.1 a)] or differential expansion type [see Figure C.1 b)] shall be as shown. Horizontal types of apparatus are described here, however, vertical types of apparatus are also usable. The apparatus consists of a displacement measuring device (often a differential transformer), specimen support system (made of vitreous silica, alumina, or fine-grained isotropic graphite), temperature-controlling device (furnace/cooler), temperature-measuring device (thermocouple) and load-controlling device. The relative extension between the specimen and the specimen holder (or the reference specimen) is detected.

As specimen holder, vitreous silica is preferred for measurements from below ambient temperature to 1 000 °C. A high purity alumina shall be used for measurements from ambient temperature to 1 500 °C. For measurements exceeding 1 500 °C, a high purity fine-grained isotropic graphite shall be used as specimen holder. In that case, nitrogen inert atmosphere or vacuum shall be used. The use of vitreous silica above 800 °C may lead to its crystallization, which involves a change in the thermal expansion coefficient. If there is any evidence of such a change, replace the specimen holder by a new one.



a) Single-rod type



b) Differential expansion type

Key

1	displacement measuring device (here differential transformer)	6	push-rod
2	measuring thermocouple	7	specimen holder
3	furnace/cooler	8	control thermocouple
4	test specimen	9	reference specimen
5	load-controlling device		

Figure C.1 — Schematic representation of the measuring apparatus

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