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Refractory products — Determination of thermal expansion

Produits réfractaires — Dosage de la dilatation thermique

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Foreword

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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 33, *Refractories*.

Refractory products — Determination of thermal expansion

1 Scope

This International Standard specifies test methods for the thermal expansion of refractory products. It describes a method for determining the linear thermal expansion percentage, the linear thermal expansion curve, and the linear thermal expansion coefficient.

This International Standard includes the following three test methods for the thermal expansion of refractory products:

a) a contact method with a cylindrical test piece;

b) a contact method with a rod test piece;

c) a non-contact method.

The characteristics of these methods are shown in Annex A.

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 836, *Terminology for refractories*

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

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 836 and the following apply.

3.1
starting point temperature
T_0
starting point temperature for collecting thermal expansion results, (record ambient temperature)

3.2
reference material
materials with a known linear thermal expansion (percentage) and coefficient

3.3
lowest limit temperature
T_1
lowest temperature in the measurement range for linear thermal expansion

3.4
highest limit temperature
T_2
highest temperature in the measurement range for linear thermal expansion

3.5
linear thermal expansion
ε_i
ratio of length L_0 at a starting point temperature T_0 versus length change ΔL_i (= $L_i - L_0$) between length L_i at a certain temperature T_i and length L_0

Note 1 to entry: $\varepsilon_i = \Delta L_i / L_0$

3.6
linear thermal expansion percentage
E_i
linear thermal expansion expressed as a percentage

Note 1 to entry: $E_i = \varepsilon_i \times 100$; $E_i = \Delta L_\mathrm{i} / L_0$ multiplied by 100

3.7
linear thermal expansion curve
curve(s) between the temperature on the abscissa and the linear thermal expansion percentage on the ordinate

Note 1 to entry: There are two types of curves, a rising temperature curve and a declining temperature curve.

3.8
rising temperature curve
curve concerning linear thermal expansion changes caused by rising temperature, which is normally called the linear thermal expansion curve

3.9
declining temperature curve
curve concerning linear thermal expansion changes caused by declining temperature, which is used for the examination of the size change of sample after heating

3.10
average linear thermal expansion coefficient
$\alpha_{T_2-T_1}$

ratio of length change $\Delta L(= L_2 - L_1)$ of a specimen within a temperature interval to that temperature interval $\Delta T(= T_2 - T_1)$, related to the length L_0 at the starting point temperature

Note 1 to entry: That means $\alpha_{T_2-T_1} = \Delta L / \left(L_0 \Delta T\right)$. The sample lengths L_1 and L_2 are at the temperatures T_1 and T_2, respectively. The unit of this value is °C^{-1}.

3.11
linear thermal expansion coefficient
α_{T_i}
value of average linear thermal expansion coefficient, $\Delta L / (L_0 \Delta T_i)$ when $\Delta T (=T_2 - T_1)$ approaches zero

Note 1 to entry: This means the slope of the tangent line on the relational line between linear thermal expansion $\varepsilon_i = \Delta L_\mathrm{i} / L_0$ at a certain temperature T_i and the temperature T_i. The unit of this value is °C^{-1}.

3.12
reference sample
substance of which the linear thermal expansion rate and coefficient of linear thermal expansion are known

Note 1 to entry: The shape of the reference sample should be the same as that of test piece.

3.13
difference of elongation
difference in length between the test piece and the reference sample of the same length as that of test piece when heated from the lower limit temperature to the upper limit temperature

4 Contact method with cylindrical test piece

4.1 Principle

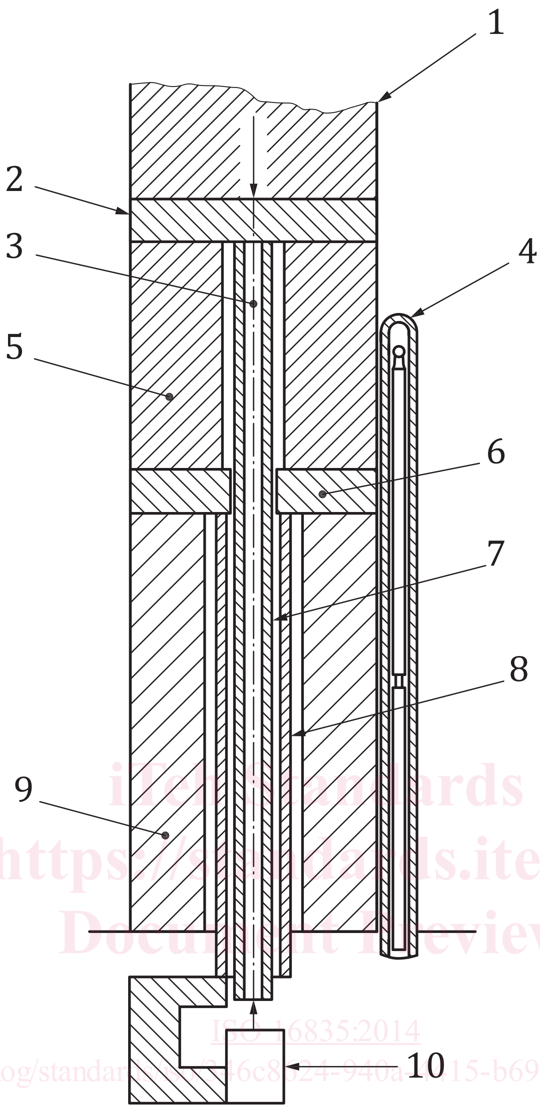
The amount of dimensional change of the cylinder test piece is continuously measured by using a contact type measurement instrument while heating at the specified rate in a heating furnace, and the linear thermal expansion rate, curve of linear thermal expansion rate, average coefficient of linear thermal expansion, and coefficient of linear thermal expansion are obtained.

4.2 Apparatus

4.2.1 Thermal expansion test apparatus

4.2.1.1 General

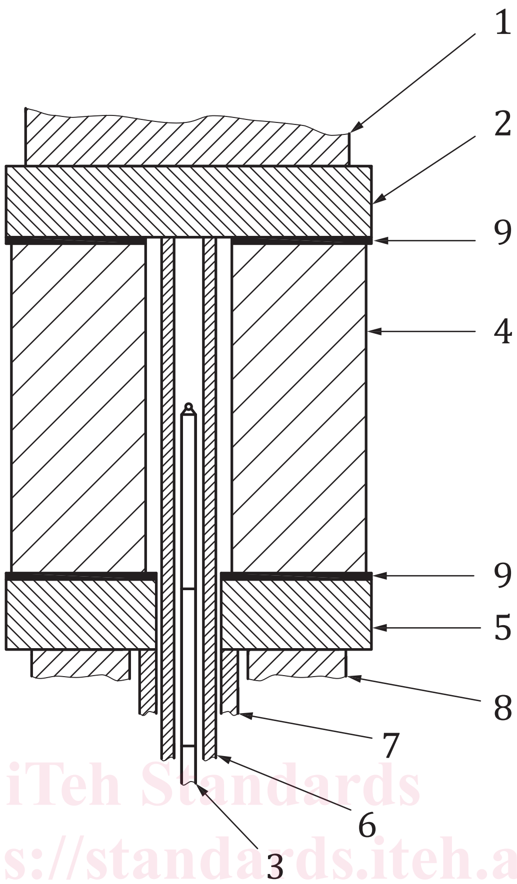
The circular pressure rod (1), test piece (5), and supporting rod (9) of the thermal expansion test apparatus shall be set in a heating furnace and all central axes aligned vertically. This alignment shall be maintained throughout the test as shown in Figure 1 and Figure 2. The structure of the apparatus shall be such that the thermal expansion of the test piece produced when a pressure of 0,01 MPa is applied to the direction of this central axis and the temperature is raised can be calculated from the relative change amount of the length of detecting tubes (7) and (8) contacted with the spacers (2) and (6) of the upper surface and the lower surface of the test piece. The contact force shall not change more than ±1 N.



Key

1 pressure rod
2 upper disk-type spacer
3 thermocouple for measuring temperature of test piece
4 thermocouple for controlling temperature of heating furnace
5 test piece
6 lower disk-type spacer
7 tube for detecting the upper position of test piece
8 tube for detecting the lower position of test piece
9 supporting rod
10 measurement instrument

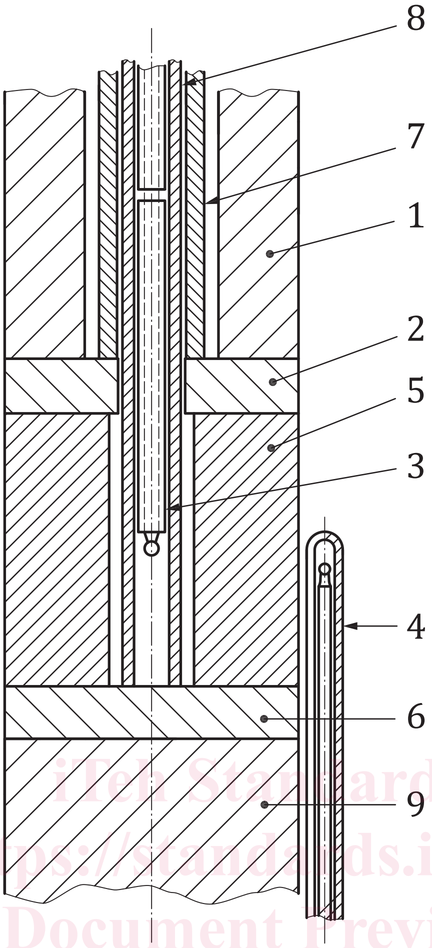
Figure 1 — Schematic drawing of the thermal expansion test apparatus (in case of measuring the change rate of test piece at the lower part of apparatus)



Key

1 pressure rod (outside diameter: 45 mm or over)
2 upper disk-type spacer (outside diameter: 50,5 mm)
3 thermocouple for measuring temperature of test piece
4 test piece (outside diameter: 50 mm ± 2 mm, inside diameter: 12 mm ± 1 mm, length 50 mm ± 0,5 mm)
5 lower disk-type spacer (outside diameter: 50,5 mm, inside diameter: 10 mm)
6 tube for detecting the upper position of test piece (outside diameter: 8 mm, inside diameter: 5 mm)
7 tube for detecting the lower position of test piece (outside diameter: 15 mm, inside diameter 10 mm)
8 supporting rod (outside diameter: 45 mm, inside diameter: 20 mm)
9 platinum or platinum rhodium foil (outside diameter: 50,5 mm, inside diameter: 10 mm)

Figure 2 — Detail drawing of thermal expansion test (in case of measuring the change rate of test piece at the lower part of apparatus)



Key

1 pressure rod
2 upper disk-type spacer
3 thermocouple for measuring temperature of test piece
4 thermocouple for controlling temperature of heating furnace
5 test piece
6 lower disk-type spacer
7 tube for detecting the upper position of test piece
8 tube for detecting the lower position of test piece
9 supporting rod

Figure 3 — Schematic drawing of thermal expansion test apparatus (in case of measuring the change rate of test piece at the upper part of apparatus)

4.2.1.2 Constitution of thermal expansion test apparatus

The apparatus shall be comprised of the following.

a) Fixed pressure rod (1): The fixed pressure rod (1) shall be a cylindrical refractory material of at least 45 mm outside diameter. In the apparatus in Figure 3, the hole of concentric circle for passing through the tubes for detecting the upper and lower positions shall be provided.

   Care shall be taken so as not to contact with the hole of the upper lid of the heating furnace.

b) Supporting rod (9): The supporting rod (9) shall be a cylindrical refractory material of at least 45 mm outside diameter. In the apparatus in Figure 1 and Figure 2, the hole of concentric circle for passing through the tubes for detecting the upper and lower positions shall be provided.

c) Disk-type spacers (2) and (6): The disk-type spacers (2) and (6) shall be the refractory material inserted for preventing the test piece from adhering to (1) and (9) by fusion due to chemical reaction, for example a disk of at least 50 mm outside diameter and 5 mm to 10 mm thickness of the alumino-silicate refractory such as high temperature sintered mullite or alumina, or the basic refractory such as magnesia or spinel. The hole of concentric circle passing through (7) shall be provided at (6) in the apparatus shown in Figure 1 and Figure 2, and at (2) in the apparatus shown in Figure 3. Both ends of (1) and (9) shall be processed so as to be flat and in parallel position, and the spacers (2) and (6) contacted with it shall be processed to make them vertical to the central axis.

   When the test piece ready to react with other refractory material, such as silica especially, is measured, the foil of platinum or platinum rhodium alloy (9) of approximately 0,2 mm in thickness may be placed between the test piece and both spacers as shown in Figure 2.

d) Tube for detecting the lower position of test piece (8): The tube for detecting the lower position of test piece (8) shall be the alumina tube of which the tip penetrates the supporting rod (9) in apparatus in Figure 1 and Figure 2 or the pressure rod (1) in the apparatus in Figure 3 and is contacted with the lower disk-type spacer adhering closely to the lower surface of the test piece, and shall be capable of moving freely so as not to make contact with the supporting rod.

e) Tube for detecting the upper position of test piece (7): The tube detecting the upper position of test piece (7) shall be the alumina tube of which the tip penetrates the supporting rod (9), the lower disk type spacer (6), and test piece (5) in the apparatus in Figure 1 and Figure 2 and is contacted with the upper disk-type spacer adhering closely to the upper surface of test piece, and shall be capable of moving freely so as not to be contacted with those. In the apparatus in Figure 3, the structures of these d) and e) are reversed.

f) Material and preparation of jigs: For the jigs, the material capable of enduring the load without deformation and reaction up to the final (highest) test temperature shall be selected.

The material from which the jigs are made should have a T_1 value greater than or equal to the temperature at which the test material has a T_5 value. T_1 and T_5 are obtained according to ISO 1893:2007.

4.2.2 Heating furnace

The heating furnace shall be the tubular furnace of which the central axis conforms to the measurement system. It shall be capable of heating the test piece up to the final (highest) test temperature at the specified rate of rising temperature [see c) in 4.4.1] in the atmosphere and of heating uniformly 12,5 mm of the upper and lower sides of test piece at 500 °C or higher within ± 20 °C of the specified temperature. The uniform heating zone around the test piece shall be measured beforehand.

The up-and-down moving type or opening-closing type is recommended because they do not hinder the setting of measurement system.

4.2.3 Detector for amount of deformation of test piece

The dial gauge or differential transformer transducer connected to an automatic recorder shall be used. These are fixed to the tip of (8), the gauge head at the tip of spindle in the case of dial gauge, or the core in case of differential transformer transducer. These then contact the tip of (7) and the relative deformation amount produced by the deformation of test piece is measured. The measuring instrument shall have the sensitivity which enables the measurement to the nearest of 0,005 mm.

4.2.4 Temperature measurement apparatus

4.2.4.1 Thermocouple for measuring the temperature of test piece

The thermocouple for measuring the temperature of the test piece shall be inserted into the alumina tube (7) which penetrates the test piece so as to be capable of measuring the temperature at the centre of the test piece and shall be arranged so that the hot contact point comes to the centre of the test piece.

4.2.4.2 Thermocouple for controlling the temperature of heating furnace

For thermocouple for controlling the temperature of heating furnace, the thermocouple with protecting tube shall be used and be arranged so that the hot contact comes adjacent to the test piece (see Figure 1).

It may be arranged adjacent to the heating unit depending on the structure of the furnace.

4.2.4.3 Type and precision of thermocouple

The thermocouple shall be a platinum-platinum rhodium system and the type capable of using up to the final (highest) test temperature shall be selected. The precision of thermocouple shall be verified.

4.2.5 Callipers

The callipers of 0,05 mm minimum scale reading shall be used.

4.2.6 Reference sample

For the reference sample, the high purity sintered alumina object of the same shape as that of the test piece specified in 4.3 shall be used. The recommended values of linear thermal expansion rate and coefficient of linear thermal expansion for reference sample are shown in Annex B.

4.3 Test piece

4.3.1 Shape of test piece

The shape of test piece shall be as follows.

a) The test piece shall be concentrically cylindrical having a 50 mm ± 2 mm outside diameter, 12 mm ± 1 mm inside diameter, and 50 mm ± 0,5 mm length.

b) The test piece shall be taken so that the upper and lower surfaces are parallel and right-angled to the central axis, and both surfaces shall be ground and polished so that the difference of length measured at any two points by using callipers does not exceed 0,2 mm. The end surface of test piece shall be placed on a flat surface plate and when a square is applied to the generating line of cylindrical test piece, the deviation, *d*, between the square and the generating line shall be 0,5 mm or below (see Figure 4).