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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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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: $E_i = \varepsilon_i \times 100$; $E_i = \Delta L_i / L_0$ 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 $\varepsilon_i = \Delta L_i / L_0$ 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

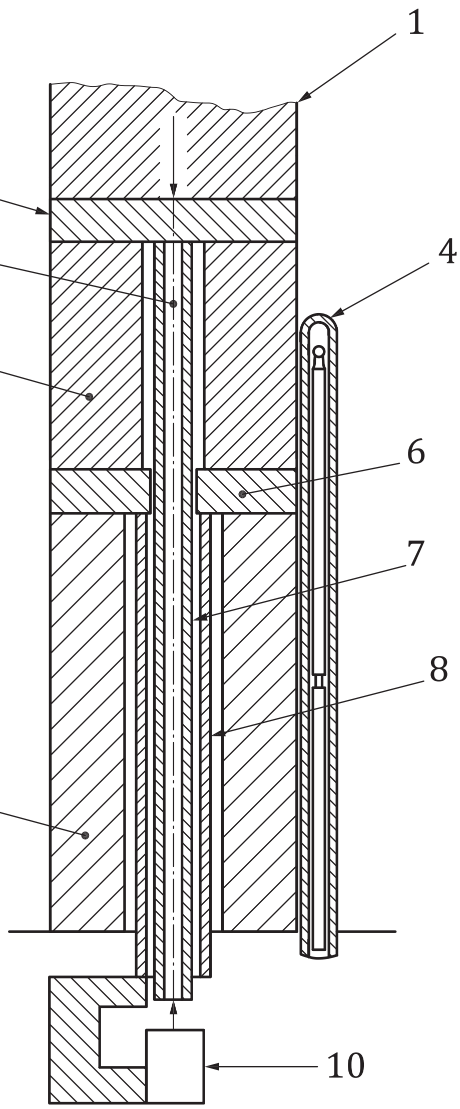
The amount of dimensional change of the cylinder test piece is continuously measured by using a contact type measurement instrument while heating at the specified rate in a heating furnace, and the linear thermal expansion rate, curve of linear thermal expansion rate, average coefficient of linear thermal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

4.2 Apparatus

4.2.1 Thermal expansion test apparatus

4.2.1.1 General

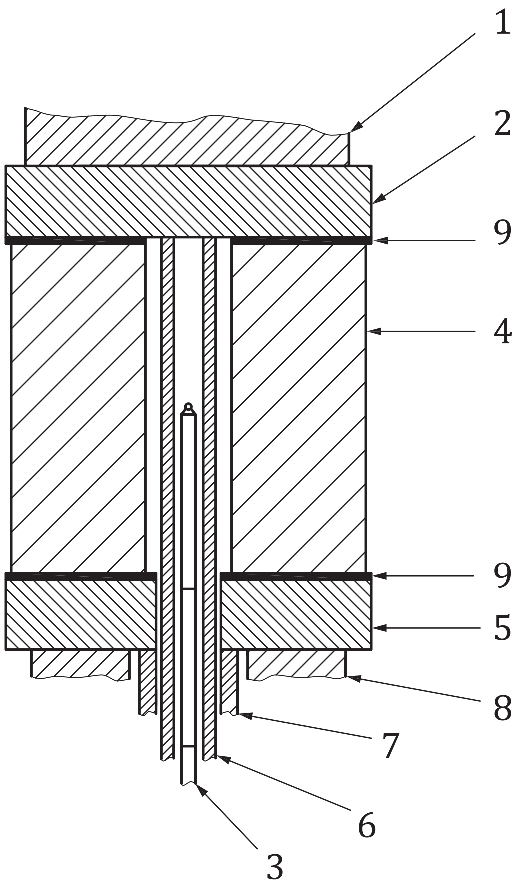
The circular pressure rod (1), test piece (5), and supporting rod (9) of the thermal expansion test apparatus shall be set in a heating furnace and all central axes aligned vertically. This alignment shall be maintained throughout the test as shown in Figure 1 and Figure 2. The structure of the apparatus 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)

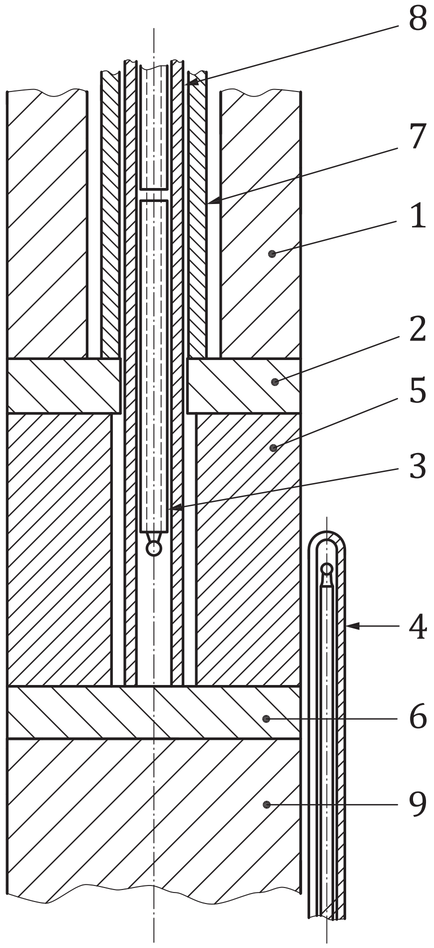
6 tube for detecting the upper position of test piece (outside diameter: 8 mm, inside diameter: 5 mm)

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.

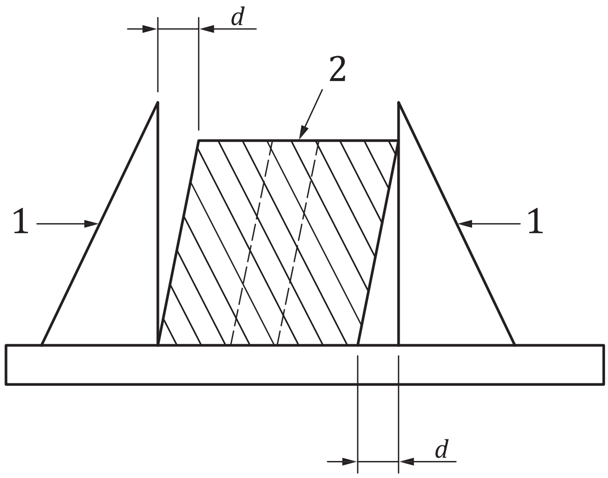
4.3 Test piece

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).



Key

1 square

2 sample

The dimension *d* is measured by using a feeler gauge.

Figure 4 — Measuring method for verticality

c) For the purpose of confirming that the upper and lower surfaces of the test piece are smooth throughout the whole surface, the filter paper of 0,15 mm thickness, upon which a carbon paper is placed, shall be placed on a surface plate and each surface shall be pressed to adhere. Both surfaces of the test piece may be coloured by using a stamp ink stand instead of carbon paper. When uncoloured part is found on the surface by this operation, it shall be polished again.

   Moreover, the smoothness of the surface may be confirmed by using a square.

4.3.2 Preparation of test piece

4.3.2.1 Shaped refractory

Unless otherwise specified, take the test sample so that the longitudinal direction of the test piece is parallel to the pressurizing direction of the sample for testing at the time of forming.

Moreover, the direction of taking the test piece may be decided according to the agreement between the parties concerned with the delivery by conforming to the purpose of using the thermal expansion result. In the case of unsintered refractory, the test piece taken from the sample after sintering at a definite temperature or the processed test piece sintered under a definite condition may be used.

4.3.2.2 Unshaped refractory

The test piece shall be either formed into the shape specified in 4.3.1 or cut off to form a definite shape. The necessity of sintering and the sintering temperature shall be subjected to the agreement between the parties concerned with the delivery. The preparation, forming, sintering condition of test piece, and the dimension of test piece shall be mentioned in the test report.

4.4 Procedure

4.4.1 Measurement of test piece

The test piece shall be measured according to the following procedure.

a) Measure the inside diameter, outside diameter, and length (to the nearest 0,1 mm) of the test piece at room temperature. Place the test piece between the supporting rod and the pressure rod with spacers in between, aligning each of their central axes.

b) Add 0,01 MPa of compression stress including the mass of pressure rod to the test piece. The change of the contact load shall not exceed ± 1 N.

   For special purposes, the load can be modified to the actual calculated load in the application field, according the agreement between the parties concerned with the delivery. In such a case, the applied compression stress should be reported.

c) Then, raise the temperature of the furnace up to the object temperature of measurement at a constant rate of 2,5 °C/min ± 0,5 °C/min.

   Moreover, in the case of the materials accompanied by volume phase transition (for example, silica and zirconia), the very slow rate of rising temperature may be used for measuring the behaviour throughout the phase transition region.

d) Measure the central temperature of the test piece by using the thermocouple (3), and record the amount of change in the direction of height of test piece at intervals within 5 min. When an abrupt change appears, record the amount of change in the direction of the height of the test piece at each temperature at intervals of 15 s.

e) If necessary, measure the relation between the temperature and length of the test piece in the cooling process.

4.4.2 Measurement of reference sample

Measure the reference sample according to 4.4.1. The reference sample shall be measured as a minimum whenever jigs are changed.

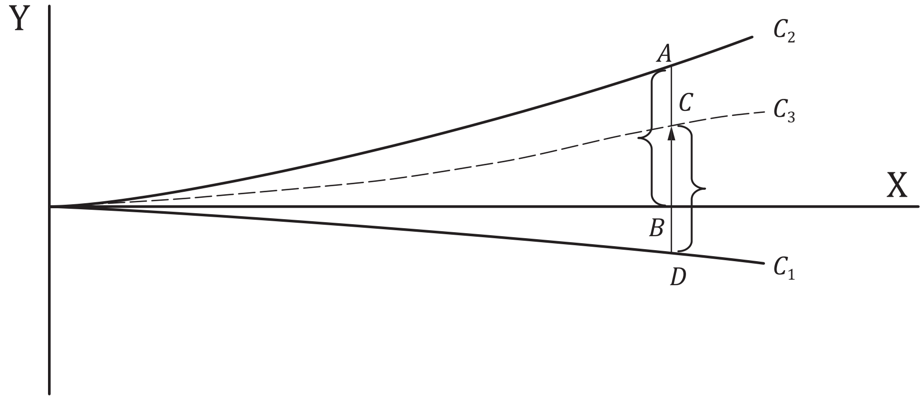
4.5 Calculation and drawing

The calculation and drawing shall be as follows.

a) The linear thermal expansion rate shall be calculated as follows.

The following steps 1) to 5) are the conceptual instructions for the correction of measured values and the calculation of linear thermal expansion rate. The actual operation should be carried out by using a computer.

1) Plot the change in the length of the test piece, without the correcting the change in the length of alumina tube for detecting position, against the temperature by using the measurement result obtained in 4.4 (curve C_1 in Figure 5).



Key

X temperature (°C)

Y change of length

Figure 5 — Correction of measurement curve

2) Measure the temperature change of length of the reference sample of the same material as that of the tube for detecting position and of the same dimension as that of the initial test piece (curve C_2 in Figure 5). Table B.2 may be used.

3) Plot the curve C_3 (= C_1 + C_2) in which curve C_1 is corrected by the curve of deformation C_2 in 2). Namely, it is AB = CD in Figure 5 at any temperature during measurement.

4) Obtain the amount of change in length of the test piece (ΔL_i) at each temperature by correcting C_3 in 3).

5) Calculate the linear thermal expansion rate according to the following formula based on the result in 4) and round off the result to two decimal places.

$$E_i = \frac{\Delta L_i}{L_0} \times 100 \quad (1)$$

where

E_i is the linear thermal expansion rate (%);

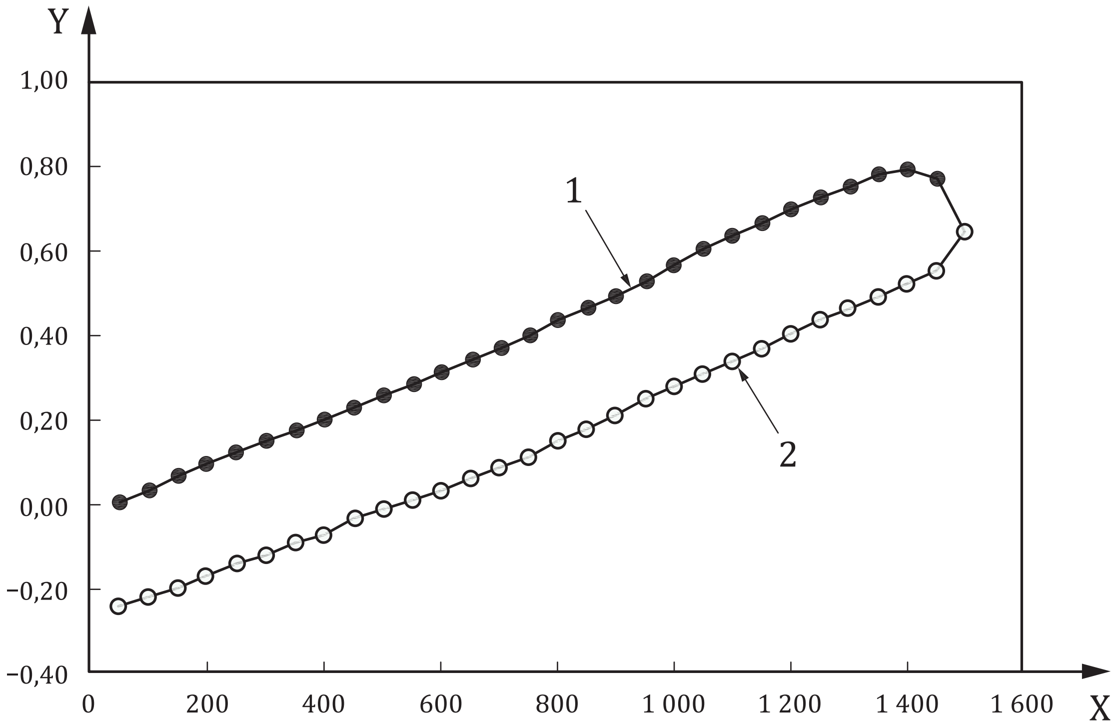
L_0 is the length of test pieces at starting temperature (mm);

ΔL_i is the amount of change in length ($L_i - L_0$) of test piece at temperature T_i (°C) (mm).

When the measured temperature is lower than the starting temperature, calculate the linear thermal expansion rate by using the value of change in length from the starting temperature. Furthermore, when the measured temperature is higher than the starting temperature, calculate the linear thermal expansion rate from the starting temperature by using the coefficient of linear thermal expansion in b) according to extrapolation method.

The starting temperature may be altered according to the agreement between the parties concerned with the delivery.

b) Obtain the curve of linear thermal expansion rate by drawing the relation between each temperature obtained in a) and its linear thermal expansion rate. An example is shown in Figure 6.



Key

X temperature (°C)

Y linear thermal expansion rate (%)

1 rising temperature curve

2 declining temperature curve

Figure 6 — Example of curve of linear thermal expansion of clayey refractory

c) Calculate the average coefficient of linear thermal expansion according to the following formula, and round off the significant figure to the place of 10^{-8}. Express the result in the unit of $10^{-6} \cdot °C^{-1}$.

$$\alpha_{T_2-T_1} = \frac{\Delta L}{L_0 \times \Delta T} \tag{2}$$

where

$\alpha_{T_2-T_1}$ is the average coefficient of linear thermal expansion of test piece (°C^{-1});

L_0 is the length of test piece at starting temperature (mm);

ΔT is the difference between lower limit temperature (T_1,) and upper limit temperature (T_2) (°C);

ΔL is the difference of elongation of test piece corresponding to temperature difference ΔT (mm).

When the relation curve between the linear thermal expansion rate and the temperature is straight throughout the whole temperature region, the upper limit temperature (T_2) can be taken as the highest temperature of measurement by taking the lower limit temperature (T_1) as the starting point. If the relation curve between the linear thermal expansion rate and the temperature is distorted, the average linear thermal expansion rate may be obtained in any temperature range as corresponding to the purpose, provided that, for indication, the average temperature range such as "$\alpha_{800\text{-}200}$" is expressed.

d) Calculate the coefficient of linear thermal expansion according to the following formula, and round off the result to two significant figures to express in the unit of $10^{-6} \cdot °C^{-1}$.

$$\alpha_{T_i} = \frac{(L_{T_{i+A}} - L_{T_{i-A}})}{L_0(T_{i+A} - T_{i-A})} \tag{3}$$

where

α_{T_i} is the coefficient of linear thermal expansion of test piece at temperature T_i (°C) (°C^{-1});

L_0 is the length of test piece at starting temperature (mm);

T_{i+A} is the temperature (A °C) higher than temperature T_i (°C);

T_{i-A} is the temperature (A °C) lower than temperature T_i (°C);

$L_{T_{i+A}}$ is the length of test piece at temperature T_{i+A} (mm);

$L_{T_{i-A}}$ is the length of test piece at temperature T_{i-A} (mm).

The recommended value of A °C is 25 °C. In case A °C cannot be taken as 25 °C, it should be taken as close to 25 °C as possible.

The relation curve between the measured linear thermal expansion and the temperature does not always become a smooth curve. Therefore, the length of test piece at (A °C) plus and minus the specified temperature T_i is obtained and taken as the coefficient of linear thermal expansion. Alternatively, the coefficient of linear thermal expansion may be determined by making the curve of the measured linear thermal expansion rate to be a function by curve fitting, and obtaining the differential value (the slope of tangent) at temperature T_i to be divided by 100.

5 Contact method with rod test piece

5.1 Principle

The rod test piece is set in a heating furnace with one of the ends fixed. The amount of dimensional change of the test piece is measured through the detecting rod while heating at a definite rate and the linear thermal expansion rate, curve of linear thermal expansion rate, average coefficient of linear thermal expansion and coefficient of linear thermal expansion at the measurement temperature, from the starting temperature, are obtained.

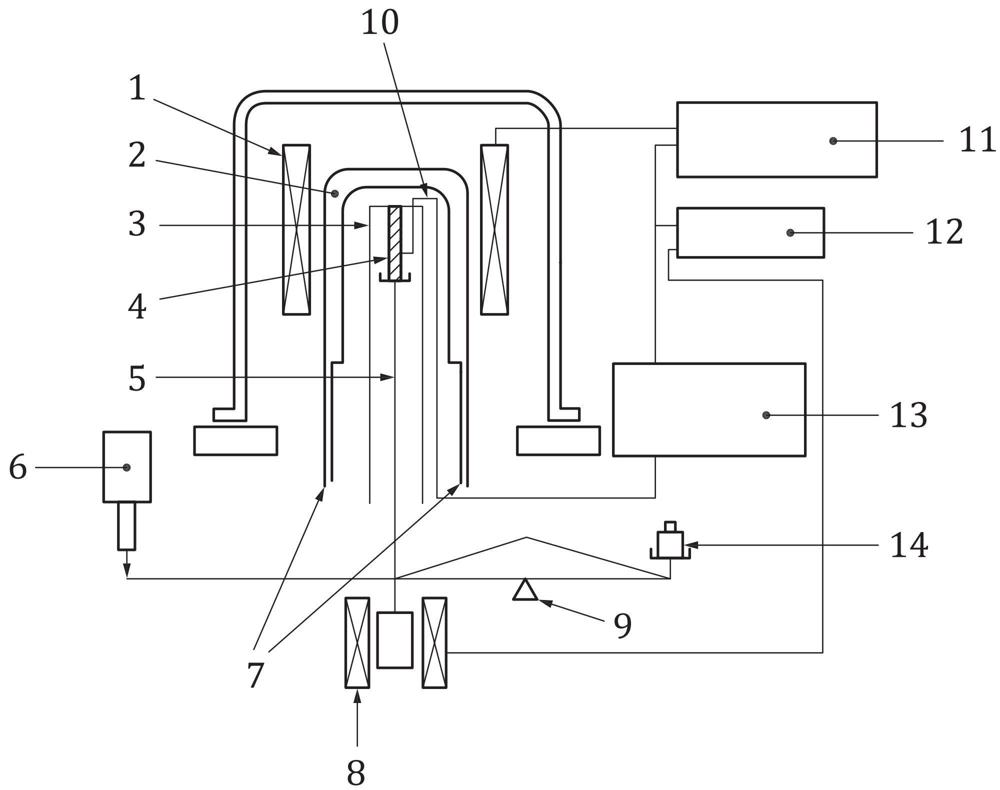
5.2 Apparatus, implement and reference sample

5.2.1 Thermal expansion test apparatus

5.2.1.1 General

The thermal expansion test apparatus is constituted by the linear thermal expansion measurement system composed of sample supporting tube, detecting rod (hereafter referred to as “supporting tube” and “detecting rod”, respectively), electric furnace (thermostatic bath), temperature control system, temperature measurement system, and recorder. The test piece may be set in either lengthwise or traverse direction. Examples of constitution of vertical type and horizontal type apparatus are shown in Figure 7 and Figure 8, respectively.

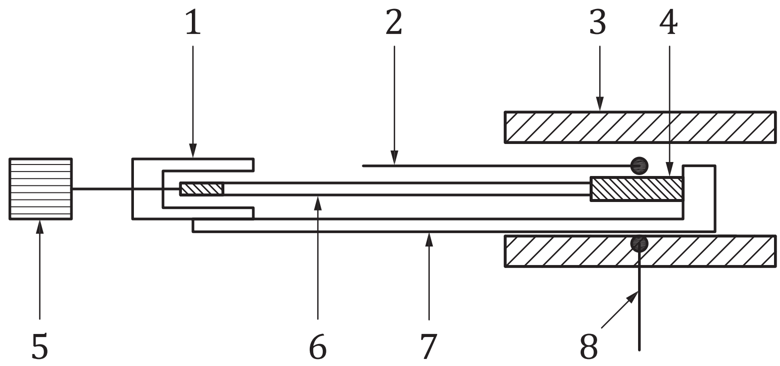
NOTE A computer can be connected to the apparatus for easier recording and processing of the results.



Key

1 electric furnace heater
2 thermostatic bath
3 supporting tube
4 test piece
5 detecting rod
6 micrometer head
7 outlet and inlet of coolant
8 linear expansion measurement system (differential transducer)
9 balance
10 thermocouple
11 temperature control system
12 recorder
13 temperature measurement system
14 weight

Figure 7 — Example of vertical type thermal expansion apparatus



Key

1 displacement measuring device

2 measuring thermocouple

3 furnace

4 specimen

5 load controlling device

6 push-rod

7 specimen holder

8 control thermocouple

Figure 8 — Example of horizontal type thermal expansion apparatus

5.2.1.2 Component of thermal expansion test apparatus

The thermal expansion test apparatus shall be comprised of the following.

a) *Supporting tube and detecting rod*: The supporting tube and detecting rod shall be all made of the same material of high purity alumina sintered object, quartz glass, or high purity graphite sintered object, and shall be the type in which the difference of coefficient of linear thermal expansion between the test piece and supporting tube, i. e. the difference of elongation due to the temperature rise between test piece and reference sample, is measured. New supporting tube and detecting rod, in order to stabilize the thermal expansion characteristic, shall be pre-sintered and cooled gradually prior to use.

   Moreover, the high purity alumina and high purity graphite shall be pre-sintered at the highest temperature to be used for approximately 7 h and then cooled gradually at the rate of 1 °C /min as a standard. The quartz glass shall be pre-sintered at 1 100 °C, for approximately 7 h and then cooled gradually down to 900 °C at the rate of 0,2 °C/min.

b) *Thermal expansion measurement system*: The thermal expansion measurement system, of which the measurement error is corrected to ± 5 × 10^{-4} % of the length of test piece (L_0) (for example, ± 0,1 µm to 20 mm in length of test piece) by using the block gauge (for example, when the length of test piece is 20 mm, 20,0 mm or 20,5 mm in approximate nominal dimension), shall be used. For the measurement of length, a differential transformer shall be used.

c) *Electric furnace (thermostatic bath):* The electric furnace shall be constructed so as to be capable of controlling heat at a definite rate and maintaining the temperature distribution of the whole heated test piece at ± 0,5 °C. For the heating unit, any one of silicon carbide heating unit, molybdenum disilicide heating unit, metal heating unit, or graphite heating unit shall be used. At least, in the electric furnace for measuring the refractory containing carbon and/or silicon carbide, the mechanism which allows an inert gas (nitrogen or argon) of a definite flow rate shall be provided.

The muffle type furnace in which the test piece is not directly subjected to the radiation heat from heating unit should be used.

For test pieces where hydrocarbon vapour is generated during heating, the apparatus should have a mechanism capable of exhausting this gas.

d) *Temperature control system*: The temperature control system shall be capable of control at ± 0,5 °C to the set value of temperature to be targeted.

e) *Temperature measurement system*: The temperature measurement system shall be capable of measuring the thermo electro motive force due to the platinum-platinum rhodium based thermocouple in the precision of ± 0,5 °C by using the electrical method of temperature measurement.

f) *Recorder*: The recorder, which is capable of recording simultaneously the difference in temperature and elongation, shall be used.

5.2.2 Micrometer or callipers

The outside micrometer or the callipers with 0,01 mm minimum graduation in scale reading shall be used.

5.2.3 Reference sample

For the reference sample, the high purity alumina sintered object or quartz glass shall be used for the measurement in atmosphere. In the atmosphere of inert gas, the high purity alumina sintered object or high purity graphite sintered object shall be used. For the supporting stand and detecting rod, the same material shall be used. The recommended values of linear thermal expansion rate and coefficient of linear thermal expansion of the reference sample are shown in Annex B.

5.3 Test piece

5.3.1 Dimension and shape of test piece

The test piece shall be either a square rod 5 mm to 12 mm in each side and 15 mm to 100 mm in length or a round rod 5 mm to 15 mm in diameter and 15 mm to 100 mm in length, processed vertically to the axial direction so that both end surfaces become parallel to each other. The dimension of each side or diameter of the section of the test piece shall be at least two times the maximum particle used in test sample and the length of test piece shall be at least four times the maximum particle. The test piece size and shape should be in agreement between the parties concerned.

Moreover, when the particle diameter used in test sample is large, it shall be measured either in non-contact method or contact method with cylindrical test piece.

5.3.2 Preparation of test piece

5.3.2.1 Shaped refractory

The test piece shall be taken, unless otherwise specified, such that the longitudinal direction of the test piece is parallel to the direction in which the test sample was pressurized in forming.

Moreover, the direction of sampling, depending on the purpose of use of the thermal expansion result, may be as agreed between the parties concerned with the delivery. In the unsintered refractory, the test piece can be either taken from the sample after sintered at a definite temperature or be supplied by sintering the processed test piece under a definite condition.

5.3.2.2 Unshaped refractory

The test piece shall be either formed into the shape specified in 5.3.1 or cut off to form a definite shape. The necessity of sintering and temperature of sintering shall be subjected to the agreement between the parties concerned with the delivery. The conditions of preparation, forming, sintering of test piece, and the dimensions of test piece shall be mentioned in the test report.

5.4 Procedure

5.4.1 Measurement of correction factor of difference of elongation

The correction factor of difference of elongation shall be calculated according to the following equation.

Measure the difference of elongation by using the test piece of the same material as that of the reference sample as the standard material according to 5.4.2 and measure the change of base line (L_b).

Measure the difference of elongation by using the high purity platinum and silicon as the standard sample according to 5.4.2 and calculate the correction factor of the difference of elongation to the high purity of platinum and silicon to two decimal places according to the following formula.

$$k = \frac{L_0 \times \Delta T \times (\alpha_{\text{ref}} - \alpha_{\text{comp}})}{L_{\text{ref}}} \tag{4}$$

where

k is the correction factor of difference of elongation of standard sample;

L_0 is the length of standard sample at starting temperature (mm);

ΔT is the difference between the lower limit temperature (T_1) and the upper limit temperature (T_2) (°C);

α_{ref} is the average coefficient of linear thermal expansion of standard sample in the range between the lower limit temperature (T_1) and the upper limit temperature (T_2) (°C^{-1});

α_{comp} is the average coefficient of linear thermal expansion of reference sample in the range between the lower limit temperature (T_1) and the upper limit temperature (T_2) (°C^{-1});

L_{ref} is the difference of elongation measured about standard sample (mm).

For the average coefficient of linear thermal expansion of standard sample and reference sample, use the values in Table 1, Table B.1, and Table B.2. Average the correction factor of the difference of elongation calculated about the high purity platinum and silicon and round off the result to two decimal places. Use this value in 5.5 as the correction factor of difference of elongation.

Table 1 — Linear thermal expansion ε and coefficient of linear thermal expansion α

Temperature °C	Platinum		Temperature °C	Silicon	
	ε (10^{-4})	α ($10^{-6}\cdot$°C^{-1})		ε (10^{-4})	α ($10^{-6}\cdot$°C^{-1})
20	0,000 0	8,9	20	0,000 0	2,5
77	0,051 3	9,1	77	0,015 6	2,9
127	0,097 2	9,2	127	0,031 0	3,2
227	0,190 9	9,5	227	0,064 0	3,5
327	0,287 1	9,7	327	0,101 0	3,8
427	0,385 6	10,0	427	0,140 0	4,0

5.4.2 Measurement of difference of elongation of test piece

The measurement of the difference of elongation shall be carried out according to the following procedure.

a) Measure the length of the test piece at room temperature using the outside micrometer or the callipers and round off the result to two decimal places.

b) Set the test piece so as not to make clearance between the supporting tube and the detecting rod and so as to stabilize and add force of 98 mN (corresponding to 10 g of weight) to the end surface of test piece.

c) Mount the thermocouple for measuring temperature such that it is near the central part of the test piece.

d) Carry out the measurement from room temperature to the specified temperature.

Moreover, unless otherwise specified, carry out the measurement to 1 500 °C.

e) The rate of rising temperature of the test piece shall be a constant rate of 4 °C /min. ± 1 °C /min.

f) Record the difference in temperature and elongation in definite intervals of 10 °C or lower. Carry out recording on a recording paper by using a recorder or by using a computer.

When the reading of a thermometer and a direct reading type detector is carried out by computer, the data processing is easy. In such a case, the amount of dimensional change should be recorded in the unit of 1 °C.

g) If necessary, measure the relation between the temperature and length of test piece in cooling process, and record it.

5.5 Calculation and drawing

The calculation and drawing shall be as follows.

a) Calculate the linear thermal expansion rate according to the following formula based on the measurement results in 5.4 and round off the resultant value to two decimal places.

$$E_i = \frac{\Delta L_i}{L_0} \times 100 \quad (5)$$

where

E_i is the linear thermal expansion rate (%);

L_0 is the length of test piece at starting temperature (mm);

ΔL_i is the amount of change in length of test piece at temperature T_i (°C) (L_i-L_0) (mm);

ΔL_i is the expressed by the following formula.

$$\Delta L_i = L_{i.\mathrm{act}} - L_b + L_{\mathrm{ref}} \tag{6}$$

where

$L_{i.\mathrm{act}}$ is the measured value of displacement in length at temperature T_i in 5.4.2 f) (including the expansion of reference sample) (mm);

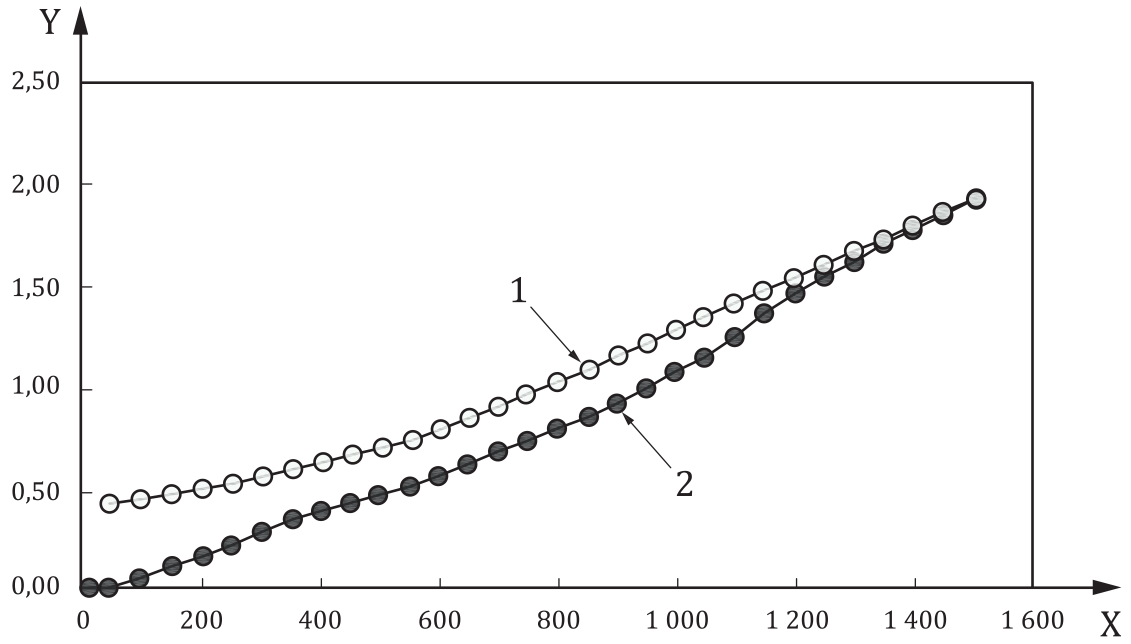
L_b is the base line value in 5.4.1 a) (mm);

L_{ref} is the displacement of length of reference sample at temperature T_i (values in Table B.1 and Table B.2) (mm).

Moreover, when the measured temperature is lower than the starting temperature, calculate the linear thermal expansion rate by using the value of change in length from the starting temperature. And when the measured temperature is higher than the starting temperature, calculate the linear thermal expansion rate from the starting temperature by using the coefficient of linear thermal expansion in c) according to extrapolation method.

The starting temperature may be altered according to the agreement between the parties concerned with delivery.

b) Obtain the curve of linear thermal expansion rate by drawing the relation between each temperature obtained in a) and its linear thermal expansion rate. An example of the curve of linear thermal expansion rate is shown in Figure 9.



Key

X temperature (°C)

Y linear thermal expansion rate (%)

1 declining temperature curve

2 rising temperature curve

Figure 9 — Example of curve of linear thermal expansion rate of magnesia carbon based refractory brick

c) Calculate the average coefficient of linear thermal expansion according to the following formula, and round off the significant figure to the place of 10^{-8}. Express the result in the unit of $10^{-6} \cdot °C^{-1}$.

$$\alpha_{T_2-T_1} = \frac{\kappa \times \Delta L}{L_0 \times \Delta T} + \alpha_{comp} \quad (7)$$

where

$\alpha_{T_2-T_1}$ is the average coefficient of linear thermal expansion of the test piece ($°C^{-1}$);

L_0 is the length of test piece at starting temperature (mm);

ΔT is the difference between the lower limit temperature (T_1) and the upper limit temperature (T_2) (°C);

ΔL is the difference of elongation of the test piece corresponding to temperature difference ΔT (mm);

κ is the correction factor of difference of elongation obtained in 5.4.1;

α_{comp} is the average coefficient of linear thermal expansion of reference sample in the range between the lower limit temperature (T_1) and the upper limit temperature (T_2) ($°C^{-1}$) (value according to Table 1).

When the curve of linear thermal expansion rate is straight throughout the whole temperature region, the lower limit temperature (T_1) may be taken as the starting point and the upper limit temperature (T_2) the highest temperature of measurement. Even if the plot of linear thermal expansion rate is nonlinear, the average linear thermal expansion rate may be obtained in any range of temperature as corresponding to the purpose, provided that, for indication, the range of temperature average is expressed such as "$\alpha_{800\text{-}200}$".

d) Calculate the coefficient of linear thermal expansion according to the following formula, and round off the result to two significant figures to express in the unit of $10^{-6} \cdot °C^{-1}$.

$$\alpha_{T_i} = \frac{(L_{T_{i+A}} - L_{T_{i-A}})}{L_0(T_{i+A} - T_{i-A})} \quad (8)$$

where

α_{T_i}	is the coefficient of linear thermal expansion of test piece at temperature (T_i) (°C) (°C^{-1});
L_0	is the length of test piece at the starting temperature (mm);
T_{i+A}	is the temperature (A °C) higher than temperature T_i (°C);
T_{i-A}	is the temperature (A °C) lower than temperature T_i (°C);
$L_{T_{i+A}}$	is the length of test piece at temperature T_{i+A} (mm);
$L_{T_{i-A}}$	is the length of test piece at temperature T_{i-A} (mm).

The recommended value of A °C is 25 °C. In case A °C cannot be taken as 25 °C, it should be taken as close to 25 °C as possible.

The relation between measured linear thermal expansion rate and temperature is not always a smooth curve. Therefore, obtain the length of the test piece at (A °C) minus and plus the specified temperature T_i and take it as the coefficient of linear thermal expansion. Alternatively, the coefficient of linear thermal expansion may be determined by making the curve of the measured linear thermal expansion rate to be a function by curve fitting, and obtaining the differential value (the slope of tangent) at temperature T_i to be divided by 100.

6 Non-contact method

6.1 Principle

A stick-shaped test piece is set up in a furnace. It is heated in the furnace with a constant rising temperature rate, the length changes of both ends of the test piece are measured at every additional interval of temperature by non-contact measuring system from outside the furnace.

Linear thermal expansion percentages, average linear thermal expansion coefficient, and linear thermal expansion coefficient are calculated from the measured length change values, the original test piece length, and the temperature change of the test piece in relation to the starting point temperature.

The linear thermal expansion curve is taken from plotting the related values between the linear thermal expansion percentage and temperature.

An example of apparatus and test piece for non-contact method is shown in Annex C. Any other facilities can be used if they satisfy the required accuracy of the measurement, with the agreement between the parties concerned.

6.2 Procedure

Procedures are as follows.

a) The length of the test piece is measured with callipers.

b) The test piece is put in measurement position in the furnace and is heated homogeneously at a rising temperature rate of 4 ± 1 °C/min to the desired temperature. During the heating process, the length changes of both ends of the test piece at each temperature are measured precisely with a direct reading detector and the relationship between the temperature of test piece and the length of test piece is recorded.

It is possible to process test results easily by thermocouple values. In this case, the length change should be recorded a 1 °C interval.

c) If necessary, in the declining process, the relationship between the temperature of test piece and the length of test piece shall be recorded.

6.3 Calculation and drawing a figure

Calculation and drawing a figure are as follows.

a) The linear thermal expansion rate is calculated with the following equation using the measurement results of 6.2 and is rounded off to the nearest second decimal places.

$$E_i = \frac{\Delta L_i}{L_0} \times 100 \qquad (9)$$

where

E_i is the linear thermal expansion percentage, in %;

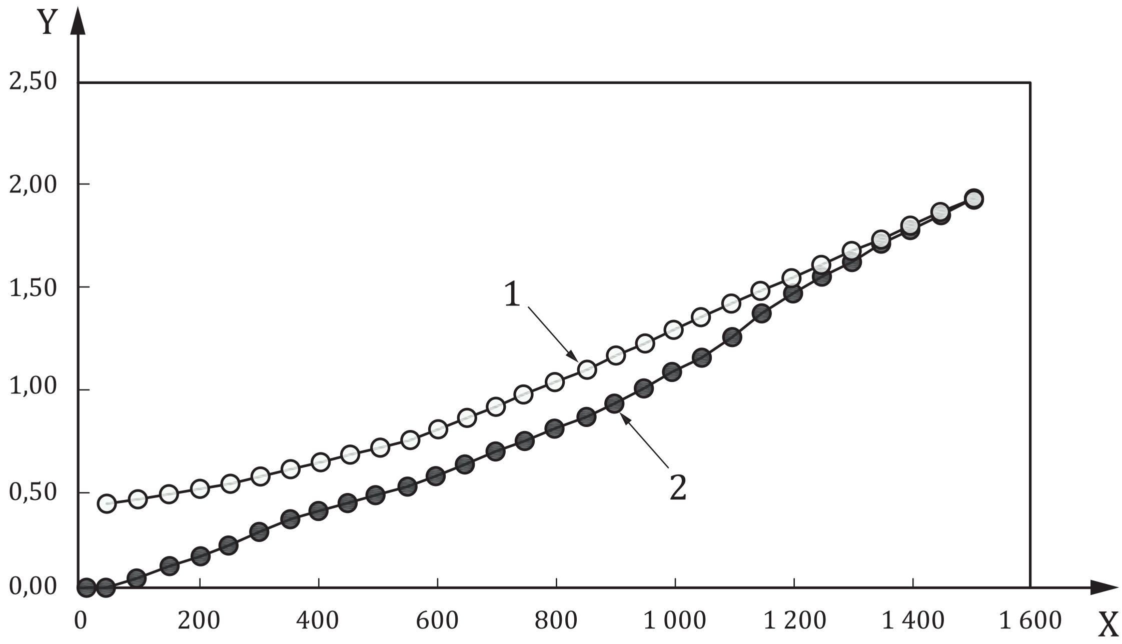
L_0 is the test piece length, in mm, at the starting point temperature;

ΔL_i is the change of test piece length $L_i - L_0$, in mm, at temperature T_i °C.

When the measured temperature is lower than the starting point temperature, the linear thermal expansion percentage is used. The results lower than starting point temperatures should be omitted. In case the measured temperature is higher than the starting point temperature, the linear thermal expansion results from the starting point temperature to the higher temperature and should be calculated by extrapolation method.

The starting point temperature should be changed as a result of the agreement between the parties concerned, if necessary.

b) The curve of the linear thermal expansion percentage is obtained by plotting the relationship between each temperature obtained in a) and the linear thermal expansion percentage. Figure 10 shows an example.



Key

X temperature (°C)

Y linear thermal expansion rate (%)

1 declining temperature curve

2 rising temperature curve

Figure 10 — Example of a linear thermal expansion curve for magnesia-carbon refractory

c) An average linear thermal expansion coefficient shall be calculated by the following equation and is rounded off to the nearest eighth decimal places and is expressed in $10^{-6} \cdot °C^{-1}$.

$$\alpha_{T_2-T_1} = \frac{\Delta L}{L_0 \cdot \Delta T} \tag{10}$$

where

$\alpha_{T_2-T_1}$ is the average linear thermal expansion coefficient of test piece, $°C^{-1}$;

L_0 is the test piece length, in mm, at starting point temperature;

ΔT is the difference, in °C, between lowest limit temperature T_1 and highest limit temperature T_2;

ΔL is the difference, in mm, of test piece length between temperature difference ΔT.

When the relationship between linear thermal expansion percentage and temperature is linear through the whole temperature range, the starting point can be the lowest temperature (T_1) and the highest temperature can be the highest limit temperature (T_2). Even if the relation line between linear thermal expansion percentage and temperature becomes nonlinear, the average linear thermal expansion coefficient is calculable in any suitable temperature range according to purpose. The average linear thermal expansion coefficient shall always express the range of temperature, e.g. "$\alpha_{800\text{-}200}$".

d) The coefficient of the linear thermal expansion is calculated by the following equation and is rounded off to the nearest eighth decimal places and is expressed in $10^{-6} \cdot °C^{-1}$.

$$\alpha_{T_i} = \frac{(L_{T_{i+A}} - L_{T_{i-A}})}{L_0(T_{i+A} - T_{i-A})} \quad (11)$$

where

α_{T_i} is the coefficient of linear thermal expansion of test piece at temperature (T_i) (°C) ($°C^{-1}$);

L_0 is the length of test piece at the starting temperature (mm);

T_{i+A} is the temperature (A °C) higher than temperature T_i (°C);

T_{i-A} is the temperature (A °C) lower than temperature T_i (°C);

$L_{T_{i+A}}$ is the length of test piece at temperature T_{i+A} (mm);

$L_{T_{i-A}}$ is the length of test piece at temperature T_{i-A} (mm).

The recommended value of A °C is 25 °C. In case, A °C cannot be taken as 25 °C, it should be taken as close to 25 °C as possible.

The relation between measured linear thermal expansion rate and temperature is not always a smooth curve. Therefore, obtain the length of the test piece at (A °C) minus and plus the specified temperature T_i and take it as the coefficient of linear thermal expansion. Alternatively, the coefficient of linear thermal expansion may be determined by making the curve of the measured linear thermal expansion rate to be a function by curve fitting, and obtaining the differential value (the slope of tangent) at temperature T_i to be divided by 100.

7 Test report

The test report shall include the following information:

a) name of the establishment where the test is carried out;

b) date of test;

c) number of this International Standard (ISO 16835) and the method used for the measurement, written in Clause 1;

d) name of measured sample (name of manufacturer, kind, type, batch number, etc.);

e) shape and dimensions of test piece;

f) preparation condition of test piece (conditions in 4.3.2 or 5.3.2, relation to the pressurizing direction at the time of forming, position when taking sample, etc.);

g) type of apparatus used and kind of displacement detector;

h) atmosphere inside the furnace (in the case of gas flowing, kind of gas and flow rate per unit time);

i) heating conditions (kind of heating unit, rate of rising temperature, rate of cooling, etc.);

j) the contact pressure used, if other than 0,01 MPa, in contact method with cylindrical test piece;

k) test result (measurement result to be required according to the agreement between the parties concerned with the delivery, among linear thermal expansion rate, curve of linear thermal expansion rate, average coefficient of linear thermal expansion, and coefficient of linear thermal expansion at each temperature);

l) abnormal phenomena observed during test;

m) information of reference material used to calibrate the apparatus;

n) IF platinum foil is used.

Annex A
(informative)

Characteristics of the test methods

Table A.1 — Characteristics of the test methods

Classification of application	Contact method using cylinder test piece	Contact method using rod test piece	Non-contact method
Refractory products constituted by small particles	A	A	A
Refractory products containing large particles	A	C	A
Refractory products easy to soften	C	C	B
Measurement with loading	A	B	D
Measurement without loading	D	D	A
A: Most suitable, B: Applicable, C: Applicable depending on purpose, D: Not applicable.			

Annex B
(informative)

Recommended values of linear thermal expansion rate and coefficient of linear thermal expansion of reference sample

This Annex specifies the recommended values of linear thermal expansion rate and coefficient of linear thermal expansion of reference sample to be used for the thermal expansion test apparatus.

B.1 Recommended values of linear thermal expansion rate and coefficient of thermal expansion of fused quartz glass

The recommended values of linear thermal expansion rate and coefficient of linear thermal expansion of fused quartz glass are shown in Table B.1.

Table B.1 — Recommended values of linear thermal-expansion rate and coefficient of linear thermal expansion of fused quartz glass

Temperature	Linear thermal expansion rate	Coefficient of linear thermal expansion	Temperature	Linear thermal expansion rate	Coefficient of linear thermal expansion
T_i	E_i	α_{T_i}	T_i	E_i	α_{T_i}
(°C)	(10^{-4})(%)	$(10^{-6} \cdot C^{-1})$	(°C)	(10^{-4})(%)	$(10^{-6} \cdot C^{-1})$
20	0	0,48	287	159	0,61
25	2,5	0,49	327	183	0,59
47	13,5	0,53	367	206	0,56
67	36	0,56	407	228	0,54
87	47,5	0,58	447	249	0,51
107	24,5	0,60	487	269	0,49
127	59,5	0,61	527	288	0,47
147	72	0,62	567	307	0,44
167	85	0,63	607	324	0,42
187	97	0,63	647	340	0,40
207	110	0,63	687	356	0,38
227	122	0,63	727	371	0,37
247	135	0,62			
In the case of using the linear thermal expansion ε_i , these should be obtained by dividing E_i by 100.					

B.2 Recommended values of linear thermal expansion rate and coefficient of linear thermal expansion of alumina

The recommended values of linear thermal expansion rate and coefficient of linear thermal expansion of alumina are shown in Table B.2.

Table B.2 — Recommended values of linear thermal expansion rate and coefficient of linear thermal expansion of alumina

Temperature	Linear thermal expansion rate	Coefficient of linear thermal expansion	Tempera-ture	Linear thermal expansion rate	Coefficient of linear thermal expansion
T_i	E_i	α_{T_i}	T_i	E_i	α_{T_i}
(°C)	(%)	$(10^{-6} \cdot C^{-1})$	(°C)	(%)	$(10^{-6} \cdot C^{-1})$
20	0,00 0	5,30	727	0,556 0	9,09
27	0,003 7	5,40	827	0,648 5	9,34
77	0,032 4	6,08	927	0,743	9,59
127	0,064 2	6,64	1027	0,840	9,85
177	0,098 6	7,10	1127	0,940	10,09
227	0,135 0	7,46	1227	1,042	10,31
277	0,173 0	7,75	1327	1,146	10,51
327	0,212 5	7,99	1427	1,252	10,67
377	0,253 0	8,18	1527	1,360	10,84
427	0,294 0	8,35	1627	1,469	11,05
527	0,379 0	8,62	1727	1,581	11,37
627	0,466 5	8,86			
In the case of using the linear thermal expansion ε_i, these should be obtained by dividing E_i by 100.					

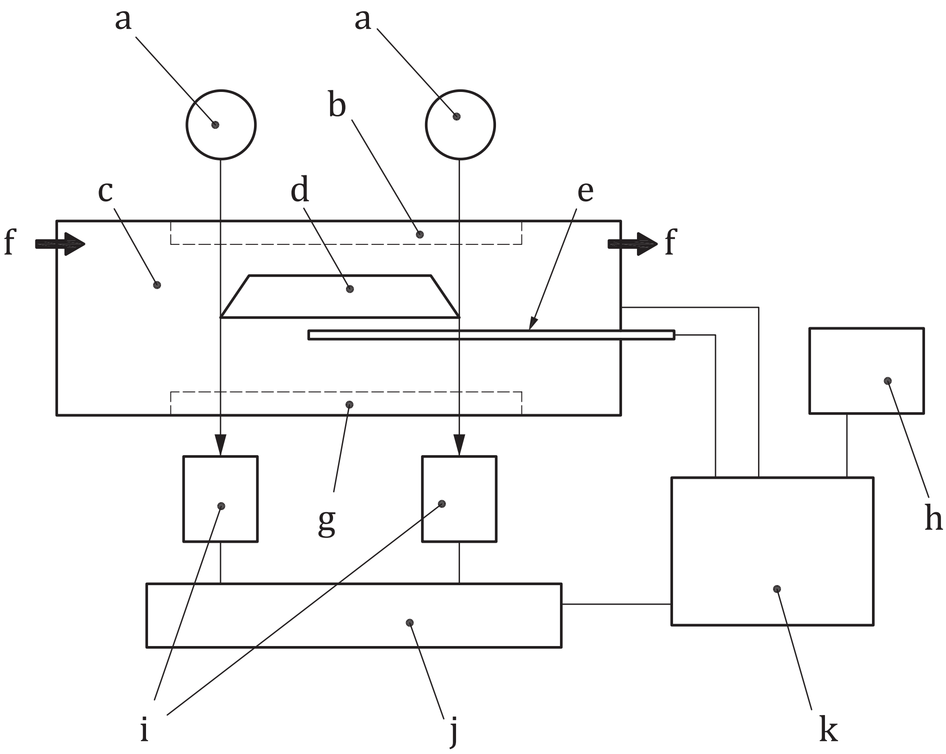
Annex C
(informative)

Example of apparatus and test piece for non contact method

C.1 Apparatus and reference material

C.1.1 Thermal expansion test instrument

The thermal expansion test instrument is composed of a heating furnace, a sample supporting stand displacement detector, and so on. A conceptual diagram of a non-contact measuring instrument is shown in Figure C.1 and Figure C.2.



Key

a light source(incandescent light)

b transparent quartz glass window

c electric furnace

d test piece

e thermocouple and temperature controller

f inert gas (if necessary)

g transparent quartz glass window

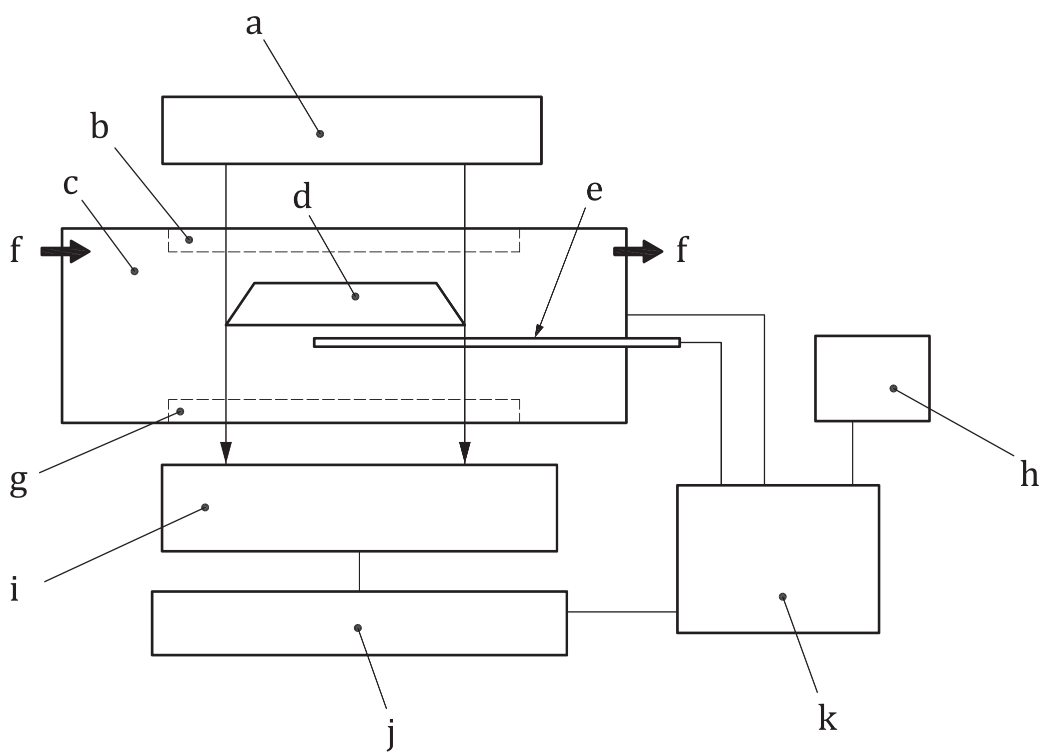
h printer

i photodetector(semiconductor detector)

j exchanger to sample position

k computer

Figure C.1 — Conceptual diagram of non-contact measuring instrument - visible light project method



Key

a laser light source

b transparent quartz glass window

c electric furnace

d test piece

e thermocouple and temperature controller

f inert gas(if necessary)

g transparent quartz glass window

h printer

i laser detector

j exchanger to sample position

k computer

Figure C.2 — Conceptual diagram of non-contact measuring instrument - infrared laser project method

C.1.1.1 Electric furnace

The electric furnace is composed of a heating element, a temperature controller instrument, a sample supporting stand, an observation window, a sample removing mechanism, and so on. The heating furnace is a horizontal tube or box shaped furnace which can heat until the final test temperature at a stipulated elevation speed [see b) in 6.2]. The electric furnace for the measurement of refractories containing carbon and/or silicon-carbide must have the flow control mechanism which keeps inert gas (nitrogen or argon) at a constant flow rate. In order to heat the test piece homogeneously, the isothermal band of the electric furnace shall be more than one and a half times area of the test piece stipulated in C.2.2.

It is desirable for a muffle furnace in which the radiant light of the heating element does not irradiate the test piece directly to be used.

If hydrocarbon vapour is generated by heating, the furnace shall have a discharge mechanism for these gases.

a) *Heating element*: A heating element of silicon-carbide, molybdenum-silicide, metal, or graphite shall be used.

b) *Observation window*: The furnace may have two windows to measure the positions of both ends of the test piece from outside. The window material must be transparent quartz glass. The furnace should have window cooling and gas curtain functions to prevent overheating of the window and to prevent fogging for generated gases.

   Distortion of the light by the window materials shall be avoided.

c) *Thermocouple for temperature measurement of sample*: A thermocouple with protective tube shall be used and its contact point shall be set up close to a test piece.

   In accordance with the structure of the furnace, a thermocouple for temperature control may be set up close to the heating element in the furnace aside from the thermocouple for temperature measurement of sample.

d) *Types of thermocouples and accuracy*: A thermocouple of platinum-platinum rhodium system stipulated by IEC 60584-1 shall be used according to the final test temperature. Thermocouple accuracy is examined by the methods prescribed in IEC 60584-2.

If a thermocouple other than the thermocouple stipulated in IEC 60584-1 is used for a measurement at high temperature, the thermocouple should be examined by the methods prescribed in IEC 60584-2.

C.1.1.2 Sample supporting stand

The sample supporting stand shall be made of a material which does not generate melting substances on surfaces contacting the test piece at high temperature. That is, the material of the sample supporting stand must not affect the measurement directly or non-directly. To restraint the high position fluctuation of the test piece, the sample supporting stand shall be as low as possible and made of material with low thermal expansion.

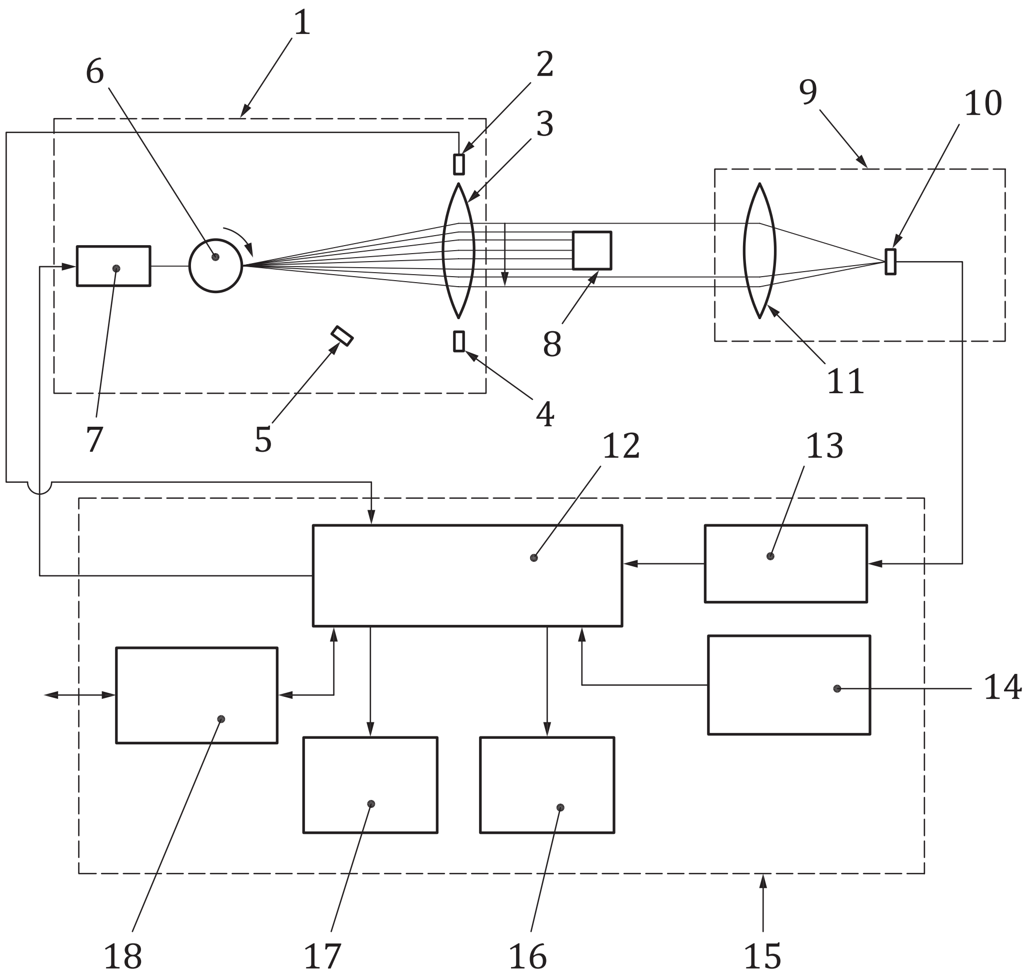
C.1.1.3 Displacement detector

The displacement detector shall be composed of a light source and a photodetector. The detector shall be able to covert the length change of both ends of a test piece by the photodetector to the size displacement of the test piece. The displacement detector has automatic and manual methods. In case of automatic methods, there is the visible light project method (Figure C.1) in which a white light is irradiated and detected by a solid-state image sensing device, and the laser projection method (Figure C.2).

Figure C.3 shows an example of a laser detector structure. Visual observation and manual detection methods include a manual comparator method where both ends of a test piece are detected by telescope and the displacement is read by a comparator and a projecting photograph method to read the displacement.

The detection resolving power of the detector shall not exceed 0,1 µm and the measurement frequency of displacement shall be within 5 °C intervals.

Introduction (insertion) of a black plate with a narrow slit between the glass window of a furnace and the photo detector is recommended to reduce detecting error of elongation due to radiation from an electric furnace.



Key

1 transmitter	10 receiving photodiode
2 photodiode for timing	11 receiving lens
3 collimation lens	12 signal processor / data processor
4 photodiode for timing	13 wave form processor
5 laser diode	14 measuring condition
6 polygon mirror / motor	15 display unit
7 motor driver	16 measuring condition display
8 test piece	17 measuring value display / alarm display
9 receiver	18 communication (RS-232C)

Figure C.3 — An example of laser detector structure

C.1.2 Slide callipers

The slide callipers with 0,01 mm minimum accuracy shall be used.

C.1.3 Reference material

As reference materials, high-purity alumina, quartz glass and/or high-purity graphite with known linear thermal expansion percentage and known linear thermal expansion coefficient shall be used.

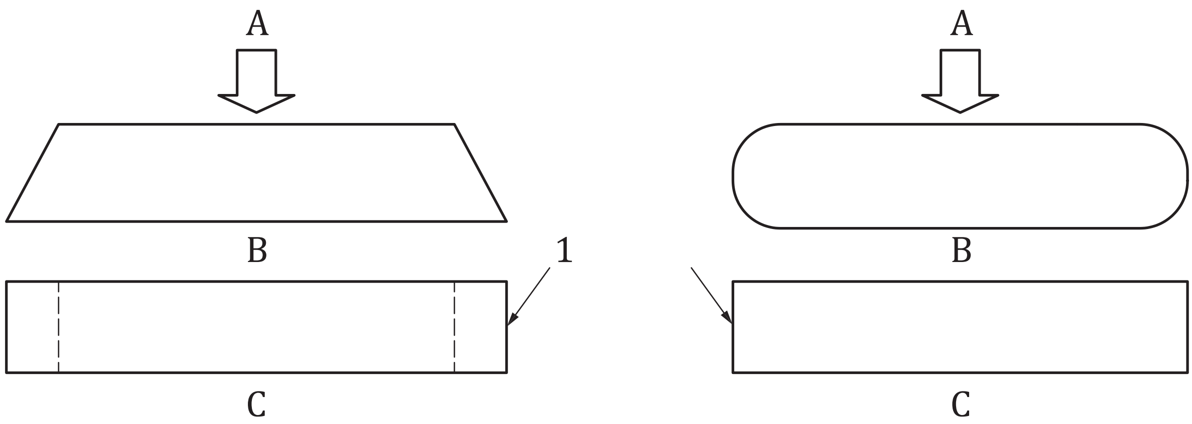
The measurement accuracy should be checked regularly using reference material.

C.2 Test piece

C.2.1 Size and shape of test piece

The test piece is a square rod 15 mm to 25 mm in diameter and 60 mm to 150 mm in length, or a cylinder rod of 15 mm to 25 mm in diameter and 60 mm to 150 mm in length. The height axis parallelism of both ends in the length direction shall be treated within parallelism ± 0,025 mm.

As shown in Figure C.4, both ends of the test piece shall be either cut sharply in a taper or rounded. The light direction shall be changed according to the measurement position. And the faces on both side shall be trimmed so that they are parallel and smooth.



a) An example of edge missing test piece

b) An example of round edged test piece

Key

A light direction

B plain view

C side view

1 smooth trimming

Figure C.4 — Shape examples of test pieces

C.2.2 Preparation methods for test pieces

C.2.2.1 Shaped refractories

The length of the test piece shall be perpendicular to the direction of the pressing during the forming of the brick, when there is no agreement on the direction of test piece with the user.

In addition, the direction of the test piece should be decided by the parties concerned according to the purposes of the thermal expansion results. In the case of unsintered refractories, test piece should be prepared after sintering at a certain temperature or a treated test piece should be sintered under specific conditions (temperature, time and so on) so it can be used as a test piece.

C.2.2.2 Unshaped refractories

The test piece shall be formed by the moulding method or a target form is prepared by cutting or drilling from a large sample obtained by moulding. The requirements for burning and burning temperature should be decided as a result of the agreement between the parties concerned. The conditions of the preparation of test pieces, moulding, burning, and size of test piece shall be recorded in the test report.

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1) This has been revised with ISO 13385-1:2011.

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