INTERNATIONAL STANDARD

ISO 1893

Third edition
2007-09-15

Refractory products — Determination of refractoriness under load — Differential method with rising temperature

Produits réfractaires — Détermination de l'affaissement sous charge — Méthode différentielle avec élévation de la température

Reference number
ISO 1893:2007(E)

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Foreword

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

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

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

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

ISO 1893 was prepared by Technical Committee ISO/TC 33, *Refractories*.

This third edition cancels and replaces the second edition (ISO 1893:2005), which has been technically revised. The main changes are corrections to the figures and improvements in the description of the calculation procedure given in 8.2.

Refractory products — Determination of refractoriness under load — Differential method with rising temperature

1 Scope

This International Standard specifies a method for determining the deformation of dense and insulating shaped refractory products, when subjected to a constant load under conditions of progressively rising temperature (or refractoriness under load), by a differential method. The test may be carried out up to a maximum temperature of 1 700 °C.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3599, *Vernier callipers reading to 0,1 and 0,05 mm*

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

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

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
refractoriness under load
measure of the behaviour of a refractory material subjected to the combined effects of load, rising temperature and time

4 Principle

A cylindrical test piece is subjected to a specified constant compressive load and heated at a specified rate of temperature increase until a prescribed deformation or subsidence occurs. The deformation of the test piece is recorded as the temperature increases, and the temperatures corresponding to specified proportional degrees of deformation are determined.

5 Apparatus

5.1 Loading device

5.1.1 General

The loading device shall be capable of applying a load centred on the common axis of the loading column, the test piece and the supporting column, and directed vertically along this axis at all stages of the test. The loading device consists of the items given in 5.1.2 to 5.1.4.

A constant compressive load is applied in a downward direction from above the test piece which is resting directly or indirectly on a fixed base. The deformation of the test piece is measured by a device that passes either through the loading device or through an intermediate base.

The text and Figures 1 and 2 show the measuring device passing through the base but, by interchanging the bored column and refractory plate with the unbored column and plate, the measuring device may pass through the loading device, as in Figure 3.

Although both arrangements are within the scope of this International Standard, it is preferable that the measuring device be positioned below the assembly, as shown in Figure 2. The reasons for this are outlined in Annex A.

5.1.2 Fixed column

The fixed column shall be at least 45 mm in external diameter and with an axial bore (see 5.1.5).

5.1.3 Moving column

The moving column shall be at least 45 mm in external diameter.

NOTE Arrangements can be made for the moving column to be fixed to the furnace, and the combination of furnace and column then forms the moveable loading device.

5.1.4 Two discs

The two discs shall be 5 mm to 10 mm thick, at least 50,5 mm in diameter and not less than the actual diameter of the test pieces, and shall be made of an appropriate refractory material compatible with the material under test.

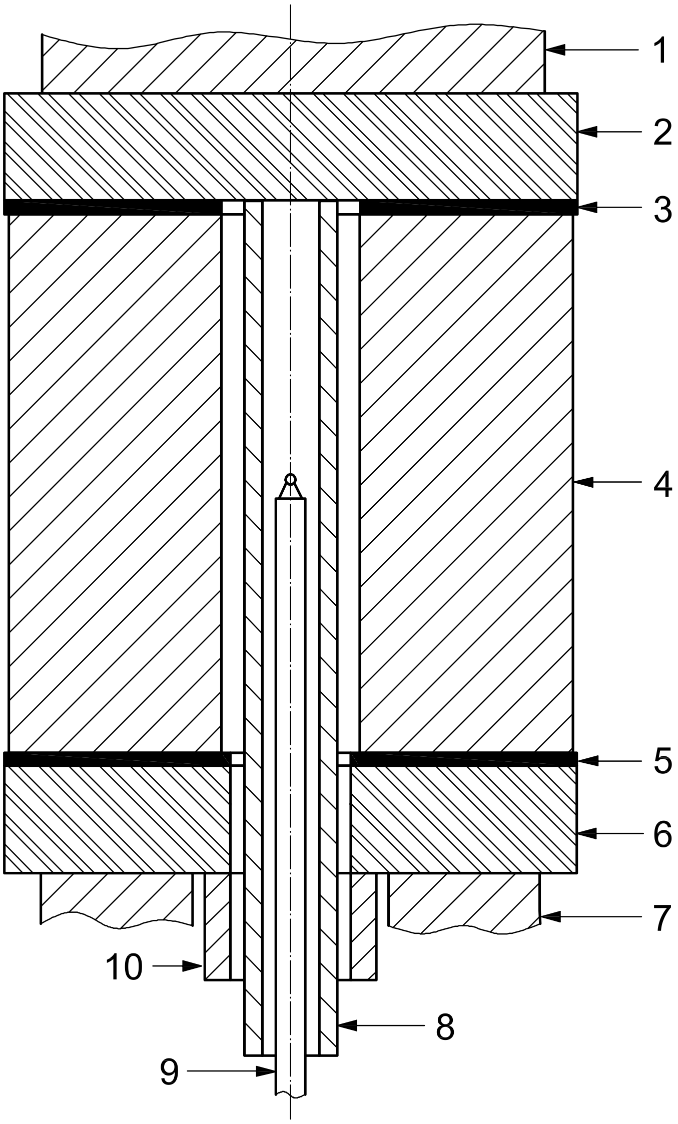
EXAMPLES High-fired mullite or alumina for alumino-silicate products or magnesia or spinel for basic products.

These discs are placed between the test piece and the fixed and moving columns. The disc placed between the test piece and the fixed column (in the arrangement shown in Figure 2) or between the test piece and the moving column (in the arrangement shown in Figure 3) shall have a central bore (see 5.1.5). The ends of the fixed and moving columns shall be plane and perpendicular to their axes; the faces of each disc shall be plane and parallel. If a chemical reaction is expected between the discs and the test piece, a platinum or platinum/rhodium foil (0,2 mm thick) shall be placed between them.

5.1.5 Layout

The arrangement of the two columns, the two discs, the platinum or platinum/rhodium foil if used, and the test piece is shown in Figure 1, which also shows typical diameters of the bores in the fixed column and the disc between them.

Dimensions in millimetres



Key

1 moving column (5.1.3), ∅ ext. 45 min.
2 upper disc (5.1.4), ∅ ext. 50,5 min.
3 Pt-Rh foil, ∅ ext. 50,5*, ∅ int. 12
4 test piece (6.1), ∅ ext. 50 ± 0,5, ∅ int. 12 min., 13 max.
5 Pt-Rh foil, ∅ ext. 50,5*, ∅ int. 10
6 lower disc (5.1.4), ∅ ext. 50,5 min., ∅ int. 10
7 fixed column (5.1.2), ∅ ext. 45 min., ∅ int. 20 min.
8 inner alumina tube (5.3.2), ∅ ext. 8*, ∅ int. 5*
9 central thermocouple (5.4.1)
10 outer alumina tube (5.3.1), ∅ ext. 15*, ∅ int. 10

NOTE Typical dimensions are marked with an asterisk (*).

Figure 1 — Example of an arrangement of test piece, columns, discs and tubes

5.1.6 Load

The columns and the discs shall be capable of withstanding the applied load up to the final test temperature without significant deformation. There shall be no reaction between the discs and the loading system. The material from which the discs are made shall have a T_1 value greater than, or equal to, the temperature at which the test material has a T_5 value (see 8.5).

5.2 Furnace

A furnace (preferably with its axis vertical) shall be used, capable of raising the temperature of the test piece to the final test temperature at the specified rate (see 7.3) in an atmosphere of air. The temperature of the region of the furnace occupied by the test piece, when at a stable temperature above 500 °C, shall be uniform around the test piece (12,5 mm above and below) to within ± 20 °C; this shall be verified by carrying out tests using the thermocouples located at different points on the curved surface of the test piece.

The furnace design should be such that the whole of the column assembly can be easily reached, either by movement of the supporting column or, if access into the furnace is restricted, by movement of the furnace itself. The assembly should be such that the test piece and loading column stand vertically and coaxial with the support column when unrestrained.

5.3 Measuring device

The measuring device shall consist of the items specified below.

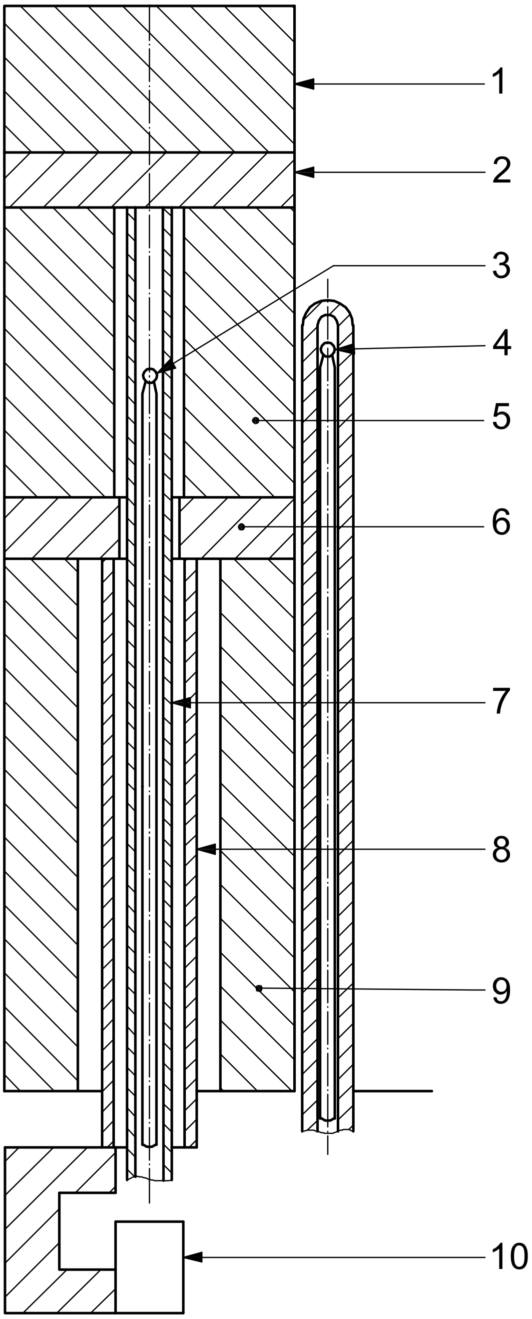
5.3.1 Outer alumina tube, placed inside the fixed column to abut on the lower side of the lower disc and free to move within the fixed column (see also 5.3.3).

5.3.2 Inner alumina tube, placed inside the outer alumina tube and passing through the bores in the lower disc and in the test piece to abut on the lower face of the upper disc, and free to move within the outer alumina tube, the lower disc and the test piece (see also 5.3.3).

5.3.3 The alumina tubes shall be capable of withstanding, without significant distortion, the load imposed on them by the measuring instrument at all temperatures up to the final test temperature.

The two possible arrangements of the two tubes, the two discs and test pieces are shown in Figure 2 and Figure 3. Where the measuring instrument is mounted above the test piece, as shown in Figure 3, adequate precautions should be taken to protect the instrument from the effects of heat rising from the furnace.

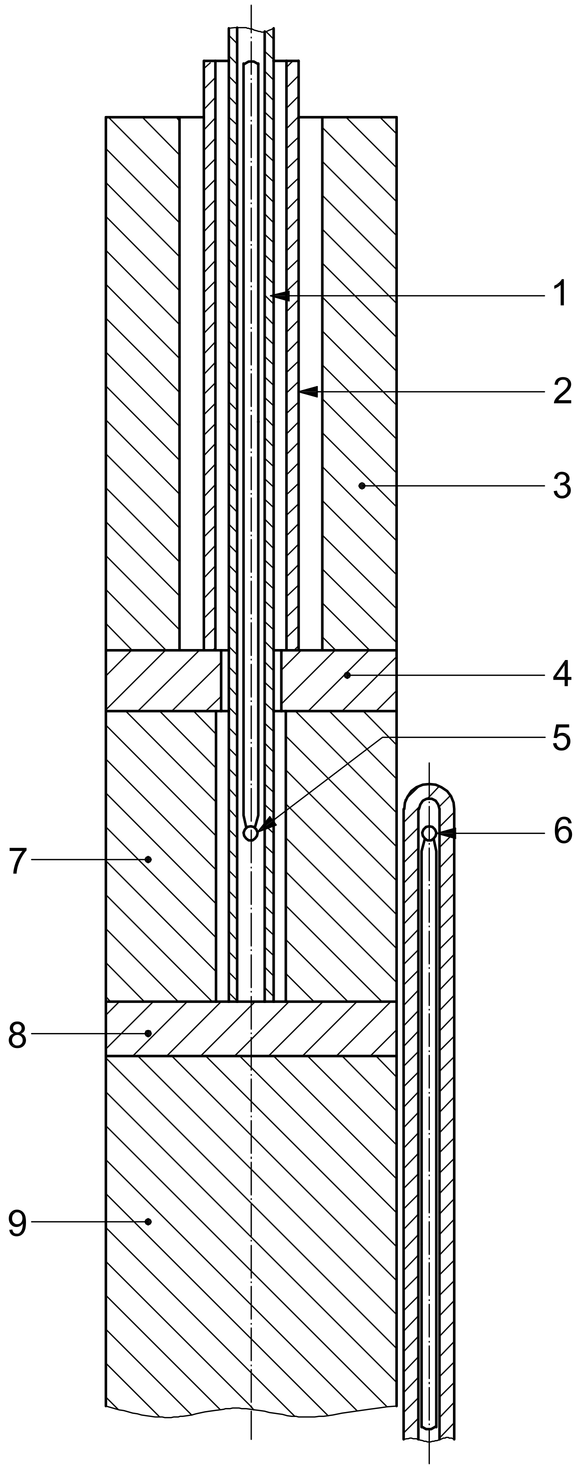
5.3.4 Appropriate measuring instrument (for example a dial-gauge or length transducer connected to an automatic recording system), fixed to the end of the outer tube (5.3.1) and actuated by the inner tube (5.3.2). The sensitivity of the measuring device shall be at least 0,005 mm.



Key

1 moving column
2 upper disc
3 central thermocouple
4 control thermocouple
5 test piece
6 lower disc
7 inner alumina tube
8 outer alumina tube
9 fixed column
10 measuring instrument

Figure 2 — Test apparatus — Measuring instrument below test piece



Key

1 inner alumina tube
2 outer alumina tube
3 moving column
4 upper disc
5 central thermocouple
6 control thermocouple
7 test piece
8 lower disc
9 fixed column

Figure 3 — Test apparatus — Measuring instrument above test piece

5.4 Temperature-measurement devices

5.4.1 Central thermocouple, passing through the inner alumina tube (5.3.2) of the dilatometer, with its junction at the mid-point of the test piece, for measuring the temperature of the test piece at its geometric centre.

5.4.2 Control thermocouple, which shall be placed in a sheath and situated outside the test piece (see Figures 2 and 3), for regulating the rate of rise of temperature.

NOTE For certain furnace constructions, it may be advisable to place the thermocouple nearer to the heating elements.

5.4.3 The thermocouples (5.4.1 and 5.4.2) shall be made from platinum and/or platinum-rhodium wire, and shall be compatible with the final test temperature. They shall be in accordance with IEC 60584-1 and IEC 60584-2. The accuracy of the thermocouples shall be checked on a regular basis.

The central thermocouple may be connected to a continuous-recording device which may form part of a temperature/displacement-recording system. In this case, calibration of the instrumentation shall be carried out regularly.

5.5 Vernier callipers

Callipers which are capable of measuring to 0,1 mm, in accordance with ISO 3599, shall be used.

6 Test piece

6.1 The test piece shall be a cylinder 50 mm ± 0,5 mm in diameter and 50 mm ± 0,5 mm in height, with a hole from 12 mm to 13 mm in diameter extending throughout the height of the test piece and bored coaxially with the outer cylindrical surface.

The axis of the test piece should preferably be in the direction in which the product was pressed.

6.2 The top and bottom faces of the test piece shall be made plane and parallel by sawing (and grinding, if necessary), and shall be perpendicular to the axis of the cylinder. All surfaces of the cylinder shall be free from visible defects. Measurements of the height at any two points, using Vernier callipers (5.5), shall not differ by more than 0,2 mm. When one face of the test piece is placed on a plane surface and a set square also in contact with the surface is brought into contact with any part of the periphery of the test piece, the gap between the side of the test piece and the set square shall not exceed 0,5 mm.

6.3 To ensure that the top and bottom ends of the test piece are flat over their entire surface, each end shall in turn be pressed on to a levelling plate which is lined with carbon paper and hard filter paper (0,15 mm in thickness). As an alternative to carbon paper, the ends of the test piece may be inked using a stamp pad. Test pieces that do not show two complete, clearly visible coloured impressions shall be reground.

It is also permissible to control the flatness of the surface with a straight edge.

7 Procedure

7.1 Measure the initial height H_0 of the test piece and measure the inner and outer diameters of the test piece to the nearest 0,1 mm. Set up the test piece between the supporting and loading columns with the spacing discs and adjust the measuring device to the correct setting. Position the assembly within the furnace.

7.2 Apply an actual load to the loading column of such magnitude that the preferred compressive stress caused in the test piece (including that due to the mass of the loading column) is as follows:

a) for dense shaped products: 0,2 MPa,

b) for shaped insulating products: 0,05 MPa,

all stresses being ± 2 %. The total load used shall be rounded to the nearest 1 N.

7.3 Raise the temperature of the furnace at such a rate that the control thermocouple (5.4.2) indicates a rate of rise of temperature of between 4,5 °C/min and 5,5 °C/min (for dense shaped products and up to a temperature of 500 °C, a heating rate of up to 10 °C/min may be used).

7.4 Record the temperature at the centre of the test piece and the readings of the measuring device at intervals of not more than 5 min throughout the test. When subsidence commences, take readings of temperature and measurement at intervals of 15 s.

7.5 Continue heating at a constant rate until the maximum permitted temperature is reached or the subsidence of the test piece exceeds 5 % of its initial height.

8 Calculation of results

8.1 Use the experimental results obtained in accordance with Clause 7 to plot the curve C_1 (see Figure 4) representing the percentage change in the height of the test piece as a function of temperature measured by the central thermocouple, uncorrected for the changes in length of the alumina tubes (5.3.1 and 5.3.2).

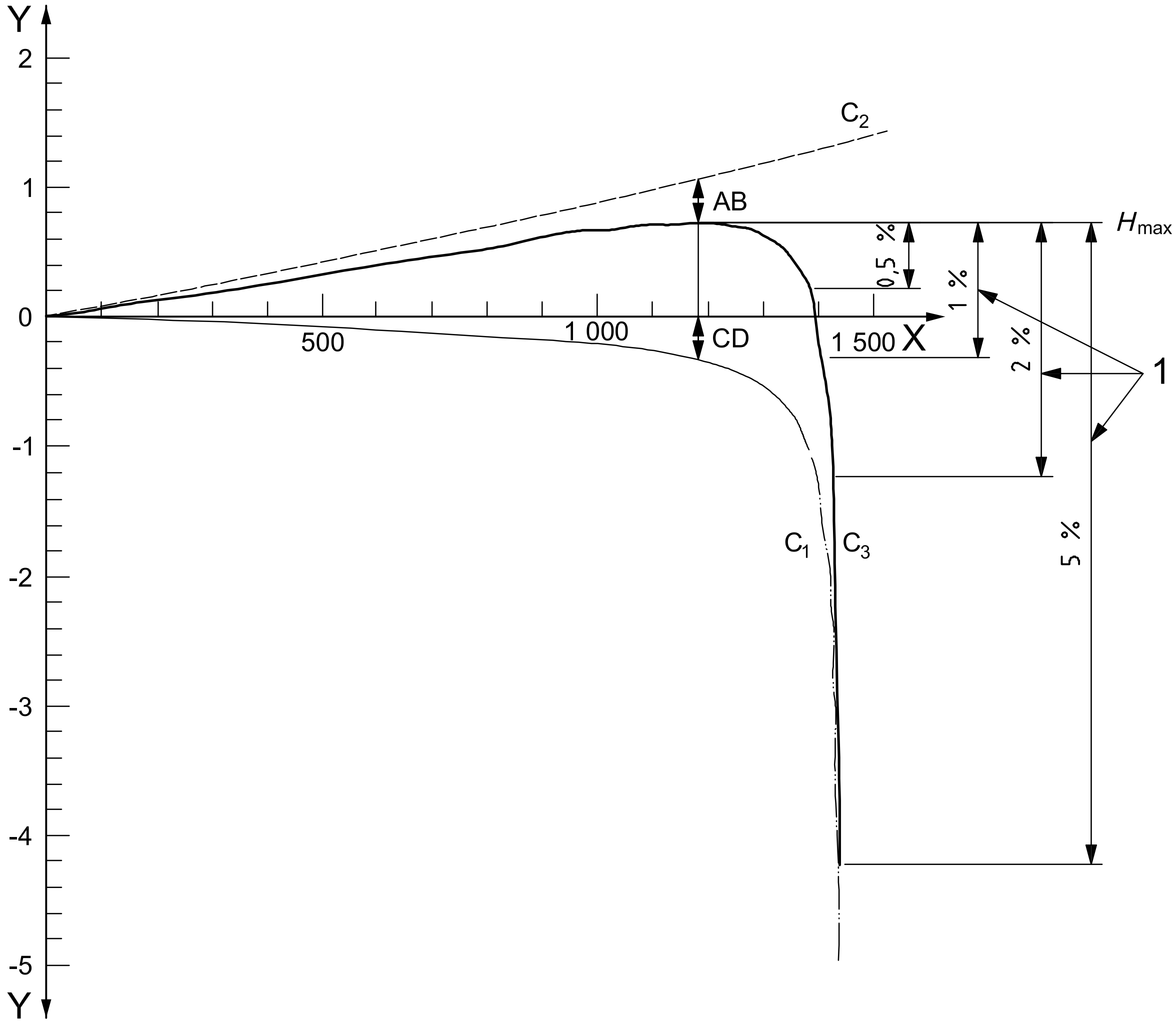
8.2 In order to determine the expansion/contraction behaviour of the test piece alone, it is necessary to compensate for the displacement caused by the expansion of the inner alumina tube. This is done by determining the expansion behaviour of a length of the inner alumina tube equal to the height of the test piece. Express the values of these changes as percentages of the initial height of the test piece and, using these percentages, plot a correction curve C_2 as shown in Figure 4.

The expansion behaviour of the alumina may be determined directly by measuring its coefficient of linear thermal expansion. Alternatively, the coefficient of linear thermal expansion specified by the manufacturer of the sintered alumina material used for the inner tube can be used to calculate this correction up to a temperature of 1 500 °C (e.g. thermal expansion at 20 °C = 0 % and at 1 000 °C = 0,82 %).

8.3 Draw the corrected curve C_3, in which, for any given temperature, the distance AB = the distance CD (see Figure 4).

8.4 Through the highest point H_{max} of this corrected curve C_3, draw a straight line parallel to the temperature axis (see Figure 4). The deformation of the test piece at a given temperature T is, by definition, the difference between the ordinate of this straight line and the ordinate of the point on the corrected curve corresponding to temperature T.

8.5 Mark on curve C_3 the points at which the deformation measured from the highest point on curve C_3 (H_{max}) in accordance with 8.4 corresponds to 0,5 %, 1 %, 2 % and 5 % of the initial height of the test piece and note the corresponding temperatures $T_{0,5}$, T_1, T_2 and T_5.



Key

X temperature, °C

Y $\Delta H/H_0$, %

1 % deformation from H_{max}

$T_{0,5}$ = 1 390 °C

T_1 = 1 405 °C

T_2 = 1 425 °C

T_5 = 1 440 °C

$C_3 = C_2 + C_1$

Figure 4 — Example of the determination of actual deformation (curve C_3) at a given temperature

9 Test report

The test report shall include the following information:

a) all information necessary for identification of the sample tested (manufacturer, type, batch number);

b) a reference to this International Standard (ISO 1893:2007);

c) details of the procedure, including:

— the position and orientation of the test piece in the original item,

— the type of furnace used,

— the nature of the atmosphere in the furnace (if other than air),

— the heating schedule and the load applied;

d) the result(s) of the test, as given by the deformation curve and the T values, determined in accordance with Clause 8 and including, if applicable, the number of tests performed on each item;

e) the name of the testing establishment;

f) any deviations from the procedure specified;

g) any unusual features observed during the test;

h) the date of the test.

Annex A
(normative)

Measuring device below or above the furnace

The arrangement of the test equipment with the measuring device below the furnace containing the test piece is preferred to the arrangement with the measuring device above the furnace for the following reasons:

a) the transducer is more easily maintained at a suitable and reasonably constant temperature;

b) the mechanical load at the hot ends of the alumina tubes (5.3.1 and 5.3.2) is kept to a minimum.

This is particularly important in the case of the inner tube (5.3.2). When the transducer is below the furnace, the load at the hot end of the inner alumina tube is equal to the force applied by the transducer spring, less the mass of the tube and the mass of the thermocouple passing through it. The force applied by the transducer spring may be set so as to be just adequate to maintain contact with the disc under all conditions.

Bibliography

[1] ISO 3187, *Refractory products — Determination of creep in compression*

ICS 81.080

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