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Refractories — Test methods for thermal shock resistance

Matériaux réfractaires — Méthodes d'essai de la résistance aux chocs thermiques

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Contents

Page

Foreword

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Refractories — Test methods for thermal shock resistance

1 Scope

This document specifies the principle, equipment, test pieces, procedures, result expression and test report of test methods for thermal shock resistance of refractories.

Three test methods are included in this document. Each one is applicable to a different product type and their test results are not comparable.

The test method, the test temperature and the test condition are intended to be negotiated by corresponding parties.

This document does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 836, *Terminology for refractories*

ISO 1927-2, *Monolithic (unshaped) refractory products — Part 2: Sampling for testing*

ISO 1927-5, *Monolithic (unshaped) refractory products — Part 5: Preparation and treatment of test pieces*

ISO 5014, *Dense and insulating shaped refractory products — Determination of modulus of rupture at ambient temperature*

ISO 5022, *Shaped refractory products — Sampling and acceptance testing*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
water quenching
rapid quenching of the fast-heated test pieces in flowing water of 5 °C to 35 °C

3.2
compressed air quenching
rapid quenching of the fast-heated test pieces in 0,1 MPa compressed air of ambient temperature

3.3
air quenching
quenching of the fast-heated test pieces in natural air of ambient temperature

4 Method 1: water quenching

4.1 General

Method 1 (water quenching) is applicable to dense aluminium silicate refractory products, but not to basic refractory products, silica refractory products, fused cast refractory products, refractory products with apparent porosity higher than 45 % or the refractories whose thermal shock resistance is hard to evaluate for low thermal shock cycles or reactions with water.

4.2 Principle

In the conditions of the specified testing temperature and water medium, the test piece with the specified shape and dimensions suffers thermal shocks. The damage degree of the hot end surface of the test piece is adopted to describe the thermal shock resistance of the refractories.

4.3 Equipment

4.3.1 Experimental furnace

Use an electric furnace whose temperature can be controlled in accordance with the specifications in 4.5. The temperature distribution of the test pieces loading area shall be uniform to ensure the temperature difference between two random points on the hot end surface is not higher than 10 °C. The uniform temperature zone shall be able to hold at least three test pieces simultaneously. One end of the thermocouple for temperature determination shall be sealed and 20 mm ± 5 mm away from the hot end surface of the test pieces. Meanwhile, the temperature record and display devices shall be equipped to control, record and display the furnace temperature continuously. A diagram of the heating device is shown in Figures 1 and 2.

4.3.2 Cooling water channel

The channel shall be able to hold multiple test pieces for rapid quenching simultaneously and ensure the inflow and outflow water temperature rise is not higher than 10 °C. A diagram of the cooling device is shown in Figures 3 and 4.

4.3.3 Test piece clamp

The clamp is used to fix test pieces for heating or cooling.

4.3.4 Drying oven

The drying oven shall be able to control the test temperature at 110 °C ± 5 °C.

4.3.5 Grid, mesh size of 5 mm × 5 mm

The grid is used to determine the hot end area of test pieces [114 mm × 64 (74) mm] in grid number.

4.4 Test pieces

4.4.1 Sampling

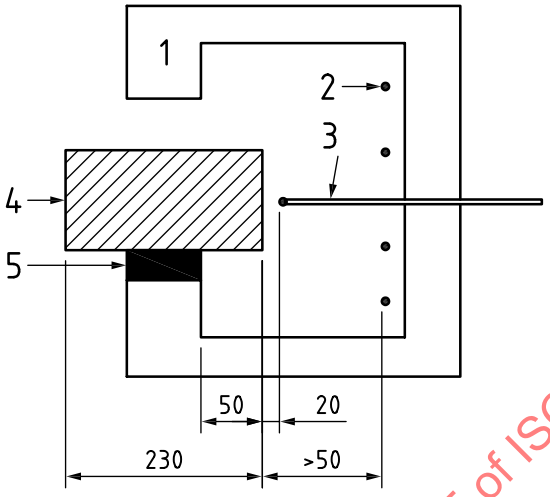
Sampling of shaped refractory products and monolithic refractories shall be conducted in accordance with ISO 5022 and ISO 1927-2, respectively, or negotiated by corresponding parties.

4.4.2 Shape, dimensions and preparation of test pieces

Standard bricks with dimensions of 230 mm × 114 mm × 64 (74) mm shall be adopted for testing.

For oversized bricks, test pieces with these dimensions shall be cut from the original bricks. One test piece for one brick only. If the test pieces of the specified dimensions cannot be obtained, the other dimensions can be negotiated by corresponding parties.

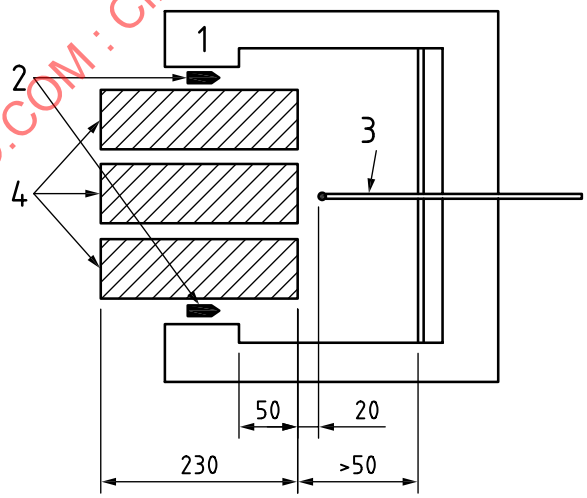
Test piece preparation of monolithic refractories shall be conducted in accordance with ISO 1927-5.



Key

1 furnace body
2 heating element
3 thermocouple
4 test pieces
5 heat insulation material

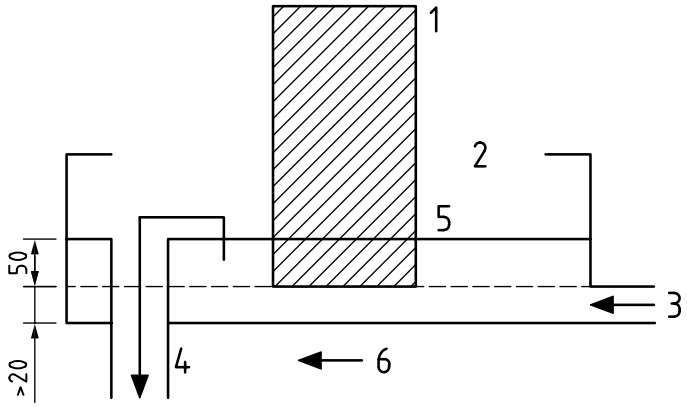
Figure 1 — Side view of heating device



Key

1 furnace body
2 heat insulation material
3 thermocouple
4 test pieces

Figure 2 — Planform for heating device



Key

1 test piece
2 water channel
3 water intake
4 water outlet
5 water surface
6 direction of water flow

Figure 3 — Side view of cooling device

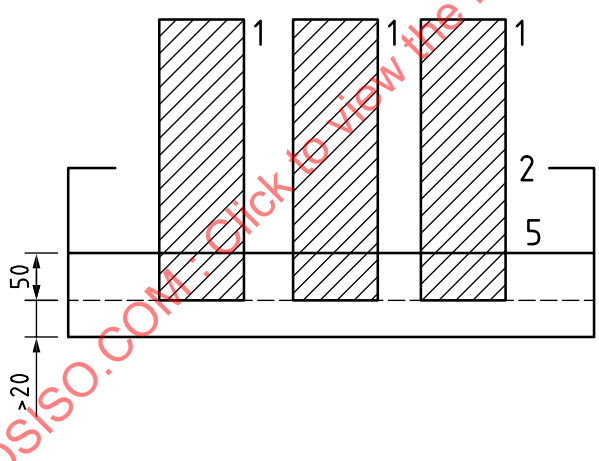


Figure 4 — Elevation view of cooling device

4.5 Test procedure

4.5.1 Heating

Dry the test pieces in an electric desiccator at 110 °C ± 5 °C to a constant mass. Clamp the test pieces with the test piece clamp at intervals no smaller than 10 mm; overlapping is not permitted. Ensure that a section with a length of 50 mm from each test piece can suffer the thermal shock. Fill the space between the clamped parts of test pieces with the insulating materials thicker than 10 mm. Measure the area of the hot end surface [114 mm × 64 (74) mm] of the test pieces in grid number A1 with a grid and record.

Preheat the furnace to the test temperature ± 10 °C and hold for 15 min. Transfer the test pieces into the hearth rapidly. The hot end surface of the test pieces shall be ≥ 50 mm away from the heating element surface. Fill the space between the test pieces and the furnace door with the insulating materials in time.

The test temperatures shall be negotiated by the corresponding parties and marked in the test report; the recommended test temperature is 1 100 °C.

After the test pieces are put into the furnace, the furnace temperature drop shall be ≤ 50 °C, and it shall return to the test temperature within 5 min. The test pieces shall be held at the test temperature for 20 min after regaining temperature.

4.5.2 Cooling

After rapid heating, take out the test pieces and immerse the hot end of the test pieces 50 mm ± 5 mm deep in the flowing water of 5 °C to 35 °C. The immersed test piece end shall be ≥ 20 mm from the water channel bottom. The temperature rise between inflow water and outflow water shall be ≤ 10 °C by adjusting the water flow rate. During quenching, close the furnace door in time to keep the furnace temperature at the test temperature ± 10 °C.

After water quenching for 3 min in the channel, take out the test pieces rapidly and leave the test pieces in air for ≥ 5 min. Measure the undamaged area of the hot end surface [114 mm × 64 (74) mm] of the test pieces in grid number A2 and record.

When the test pieces are left in air for ≥ 5 min and the furnace temperature returns to the test temperature, transfer the hot end of the test pieces into the furnace rapidly. Repeat these procedures until (50 ± 5) % of the hot end surface is damaged. Record the thermal shock cycle number or end the test at the negotiated cycles.

During the test, the mechanical damage between the test pieces and the furnace door or the water channel is not permitted.

4.6 Results expression

The thermal shock cycle in which half (or more) of the hot end surface of the test piece is damaged during water quenching is regarded as the result of the thermal shock resistance test.

The hot end surface damage rate of test pieces is calculated using the grid number of the hot end surface A_1 before the test and the grid number of the undamaged hot end surface A_2 after the test according to Formula (1).

$$P = \frac{A_1 - A_2}{A_1} \times 100\,\% \tag{1}$$

where

P is the hot end surface damage rate of the test piece, in %;

A_1 is the grid number of the hot end surface before the test;

A_2 is the grid number of the undamaged hot end surface after the test.

The damage rate shall be rounded to the nearest 1 %.

During water quenching, if half of the hot end surface is damaged this thermal shock cycle is valid.

If the test piece is damaged by external forces the test is invalid. In this case, retest.

5 Method 2: compressed air quenching

5.1 General

Method 2 (compressed air quenching) is applicable to basic refractory products, silica refractory products, fused cast refractory products and the refractories whose thermal shock resistance is hard

to evaluate for their less thermal shock cycles or reactions with water. It is also applicable to dense aluminium silicate refractory products. However, the thermal shock resistance tested by this method is not the same or comparable to that tested by the water quenching method. It is not applicable to refractory products with apparent porosity higher than 45 %.

5.2 Principle

In the conditions of the specified testing temperature and cooling medium, the test piece with the specified shape and dimensions suffers thermal shocks. At the end of each thermal shock, a rupture test with a certain bending stress (load) or a blowing burst test is conducted on the test piece. The experienced thermal shock cycle of the test piece is adopted to describe the thermal shock resistance.

5.3 Equipment

5.3.1 Experimental furnace. Use an electric furnace whose temperature can be controlled in accordance with the specifications in 5.5. The temperature distribution of the test piece loading area shall be uniform to ensure the temperature difference between two random points on the hot end surface is lower than 10 °C. The uniform temperature zone shall be able to hold at least three test pieces simultaneously. One end of the thermocouple shall be sealed. Temperature record and display devices shall be equipped to control, record and display the furnace temperature continuously.

5.3.2 Blowing device, with a nozzle of 8 mm diameter and 5 mm length.

5.3.3 A steel board (400 mm × 250 mm × 20 mm), with positional pins for test piece fixing. The position of the pins shall be proper to let the nozzle target the centre of the test piece according to the dimensions of the test piece.

5.3.4 Tester of cold modulus of rupture. The curvature radius of the contacts shall be 5 mm ± 0,5 mm. The distance between the two lower contacts shall be 100 mm ± 1 mm. The other requirements shall be in accordance with the specifications of ISO 5014.

5.3.5 Insulating pincher to hold the heated test pieces.

5.3.6 Drying oven, from room temperature to 300 °C.

5.4 Test pieces

5.4.1 Sampling

Sampling of shaped refractory products and monolithic refractories shall be conducted in accordance with ISO 5022 and ISO 1927-2, respectively, or negotiated by corresponding parties.

5.4.2 Shape, dimensions and preparation of test pieces

Rectangular test pieces with dimensions of (114 ± 3) mm × (64 ± 2) mm × (64 ± 2) mm shall be adopted. The two surfaces along the length direction shall be parallel. The allowable deviation of the parallelism between the tensed surface and the compressed surface shall not be larger than 0,5 mm and the allowable deviation of the parallelism between the two opposite edges of the middle section shall not be larger than 0,2 mm.

Cut one test piece from the working end of each brick. The working surface shall be the spraying and tension surface of the test piece. If the working surface cannot be confirmed, the original surfaces enduring the press during brick shaping shall be considered as the spraying and tension surface. Meanwhile, the surface shall be well marked. Defects caused by sampling, such as cracks and hydration, are not allowed. If they occur then resample.

Test piece preparation of monolithic products shall be conducted in accordance with ISO 1927-5.

5.5 Test procedure

5.5.1 Heating

Dry the test pieces in a drying oven at 110 °C ± 5 °C to a constant mass. Put the dried test pieces into an electric desiccator preheated to 250 °C to 300 °C and keep for at least 2 h. Preheat the furnace to the test temperature ± 10 °C and keep for 15 min. Transfer the test pieces into the hearth quickly and shut the door at once. The temperature drop of the furnace shall not exceed 50 °C. The temperature shall resume to the test temperature ± 10 °C within 5 min of putting the first test piece into the hearth. Keep this temperature for 30 min. The test pieces shall be placed on a long surface; overlapping is not permitted. The intervals between test pieces and the hearth shall be ≥ 10 mm.

The test temperatures shall be negotiated by the corresponding parties and marked in the test report. The recommended test temperature is 950 °C.

5.5.2 Cooling

Take the test pieces out and place them on a steel plate with positional pins. Make the nozzle face the centre of the blast surface of the test piece. The nozzle shall be 100 mm ± 2 mm away from the blast surface centre of the test piece. Blast for 5 min using the compressed air. The compressed air is dry and ambient temperature. The pressure before the nozzle shall be kept at (0,1 ± 0,01) MPa. Always blast the same surface.

If the test condition is the same as that in 5.5.1 and 5.5.2, several pieces can be tested simultaneously or successively.

5.5.3 Determination

After 5 min rapid quenching by compressed air, remove the test piece. Conduct the three-point bending test using the blast surface as the tension surface. Load uniformly; the maximum bending stress shall be (0,3 ± 0,01) MPa. If the test piece can endure the three-point bending test under (0,3 ± 0,01) MPa, when the furnace temperature resumes to the test temperature, transfer the test piece to the furnace quickly. Repeat these procedures until the test piece is damaged and cannot be used for the bending stress test or the test piece fractures during three-point bending test. Record the thermal cycle number or stop the test at the negotiated cycles.

The repeating thermal cycles shall be carried out continuously. If the test is interrupted, put the test pieces in a 250 °C to 300 °C desiccator. Restart the test according to these procedures. Tests for silica products are not allowed to be interrupted.

After the compressed air quenching, the time of the test pieces placed at ambient temperature is not allowed to exceed 3 min, which includes the time for the bending stress test.

Damage by external forces shall be avoided during testing.

5.6 Results expression

The thermal shock resistance is represented by the thermal shock cycle endured by the test piece before damage.

The thermal cycle in which the test piece fractures in the bending stress test or is damaged during air quenching and cannot be used for the three-point bending test is valid.

The thermal cycle in which the test piece is damaged during fast heating and cannot be used for the three-point bending test is invalid.

If the test piece is damaged by external forces, the testing is invalid. In this case retest.

6 Method 3: air quenching

6.1 General

Method 3 (air quenching) is applicable to the refractory products with apparent porosity higher than 45 %.

6.2 Principle

In the conditions of the specified testing temperature and air medium, the mass of the test piece with the specified shape and dimensions will change after thermal shocks. The mass loss rate is used to judge the thermal shock resistance of the refractory products.

6.3 Equipment

6.3.1 Experiment furnace. Use an electric furnace whose temperature can be controlled in accordance with the provisions in 6.5. Others refer to 5.3.1.

6.3.2 Manipulator or clamp, which shall be able to clamp the test pieces and put them on the cooling rack.

6.3.3 Test piece cooling rack, which shall be a steel frame spread with parallel refractory bricks with 180 mm of spacing, as shown in Figure 5.

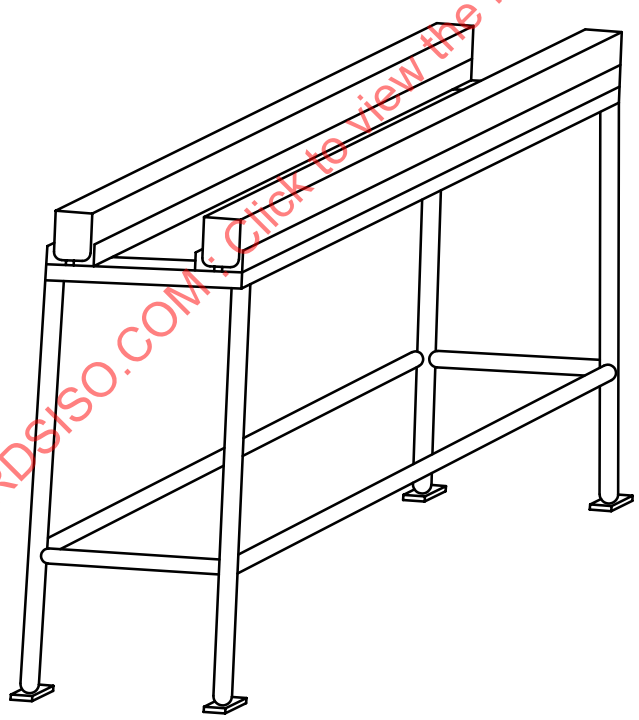


Figure 5 — Test piece cooling rack

6.3.4 Drying oven, which can control the temperature at 110 °C ± 5 °C.

6.3.5 Steel rulers.

6.3.6 Balance, with less than 5 g of precision.