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Refractory materials — Determination of thermal conductivity —

Part 1: Hot-wire method (cross-array)

Matériaux réfractaires — Détermination de la conductivité thermique —

Partie 1: Méthode du fil chaud (croisillon)

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Refractory materials — Determination of thermal conductivity —

Part 1: Hot-wire method (cross-array)

1 Scope and field of application

1.1 This part of ISO 8894 specifies a hot-wire method for the determination of the thermal conductivity of refractory products and materials.

1.2 The method is applicable at temperatures not higher than 1 250 °C and to materials whose thermal conductivity is less than 1,5 W/(m·K) and whose thermal diffusivity is less than 10^{-6} m²/s.

1.3 Subject to the limits in 1.2, the method is applicable to powdered or granular materials (but see 5.7 and 7.3).

NOTE — The thermal conductivity of bonded bricks and of prepared unshaped (monolithic) refractories may be affected by an appreciable amount of water that is retained after hardening and setting and is released on firing. These materials may therefore require pre-treatment; the nature and extent of such pre-treatment and the period for which the test piece is held at measurement temperature, as a preliminary to the performance of the test, are details that are outside the scope of this part of ISO 8894 and should be agreed between the interested parties.

1.4 The method is not applicable to refractory materials consisting of or containing fibres.

NOTE — Consideration is being given to adapting the method for use with fibrous materials.

1.5 The determination of the thermal conductivity of refractory materials by the parallel hot-wire method will be subject of ISO 8894/2.

2 Reference

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

3 Definitions

3.1 thermal conductivity, λ: Density of heat flow rate divided by temperature gradient.

The unit of thermal conductivity is the watt per metre kelvin.

3.2 thermal diffusivity, α: Thermal conductivity divided by heat capacity per unit volume.

The unit of thermal diffusivity is the metre squared per second.

3.3 heat capacity per unit volume: Heat capacity divided by volume.

The unit of heat capacity per unit volume is the joule per metre cubed kelvin.

NOTE — This is equivalent to the heat capacity per unit mass of the material multiplied by the bulk density.

4 Principle

Heating of a test piece in a furnace to a specified temperature and maintaining at that temperature. Further local heating by a linear electrical conductor (the hot wire) embedded in the test piece and carrying an electrical current of known power that is consistent in time and along the length of the test piece. Calculation of the thermal conductivity from the known power input to the hot wire and its temperature at two known intervals of time after the heating current is switched on, the variation in temperature of the hot wire being a function of the thermal conductivity of the materials of the test piece.

5 Apparatus

5.1 Furnace, capable of taking one or more test assemblies up to a maximum of 1 250 °C. The temperature at any two points in the region occupied by the test pieces shall not differ by more than 10 °C. The temperature in that region during a test (about 15 min) shall not vary by more than 0,5 °C and shall be known with an accuracy of ± 5 °C.

5.2 Hot wire, preferably of platinum or platinum/rhodium, about 200 mm in length and not exceeding 0,5 mm in diameter, the length being known to within ± 0,5 mm.

NOTE — A hot wire made of base metal is also permitted for use at temperatures below 1 000 °C.

5.3 Power supply to the hot wire, either a.c. or d.c., and not varying in power during the period of measurement by more than 2 %.

5.4 Measuring crosspiece, formed by the hot wire and the platinum/platinum-rhodium thermocouple which is welded to it at its centre. The limbs of the thermocouple shall be at right angles to the hot wire (see figures 1 and 2). The maximum diameter of the limbs of the thermocouple shall be not greater than the diameter of the hot wire (to minimize loss of heat at the measuring point by conduction).

5.5 Measuring circuits: To each end of the hot wire are welded two wires of the same type (of a diameter greater, if possible, than that of the hot wire itself), one to supply the heating current and the other for the measurement of voltage drop. The thermocouple welded to the centre of the hot wire (see 5.4) is connected in opposition to a reference thermocouple to allow the temperature changes to be measured. The wires are long enough to reach outside the furnace, where connections to the measuring apparatus may be made by wires of a different type.

5.6 Measuring apparatus (see figure 3)

5.6.1 The voltage drop between the ends of the hot wire shall be measured with an accuracy of ± 0,5 %. As an alternative to the measurement of the voltage drop, the resistance of the hot wire may be measured, with the same accuracy; if the total temperature rise exceeds 15 °C, it is necessary to allow for the variation in the resistance of the hot wire with temperature (see 7.9).

5.6.2 The current through the hot wire shall be measured with an accuracy of ± 0,5 %.

5.6.3 The apparatus for measuring the temperature of the hot wire shall have a sensitivity of 10 µV/cm, with an accuracy of 1 %.

5.7 Containers for powdered granular material, if the test is performed on such material, having internal dimensions equal to those specified for solid test piece sections in 6.2, so that the "test piece" consists of either two or three sections as in 6.2. The lower or the bottom container shall have four sides and a base, and the upper or the middle and top containers shall have four sides only.

6 Test pieces

6.1 The number of items to be tested shall be determined in accordance with ISO 5022 or another standard sampling plan.

NOTE — In this connection, if each item (specimen) is of sufficient size, so that the two or three sections that make up a test piece can be cut from it (see 6.2 and figures 1 and 2), *n* tests can be conducted from *n* specimens; if the specimens are smaller and only one section can be cut from each of them, *n* tests will require 2*n* specimens (for two-section test pieces) or 3*n* specimens (for three-specimen test pieces).

6.2 Each test piece shall consist of either two or three identical sections not less in size than 200 mm × 100 mm × 50 mm.

NOTE — It is recommended that the size of each section should be 230 mm × 114 mm × 76 mm or 230 mm × 114 mm × 64 mm. Standard size bricks may thus be used as the sections of a test piece, subject to the requirements of 6.3.

6.3 Each surface of a section of a test piece in contact with another section shall, if necessary, be ground so that the deviation from flatness between two points not less than 100 mm apart is not more than 0,2 mm.

6.4 In dense materials, when a two-section test piece is used, two straight grooves for the measuring crosspiece (5.4) and a V-groove for the reference thermocouple (5.5) shall be machined in the upper (contact) face of the lower section (see figure 2). When a three-section test piece is used, the grooves for the measuring crosspiece shall be machined in the upper face of the bottom section and the V-groove for the reference thermocouple in the upper face of the centre section (see figure 2). In either case neither the depth nor the width of the groove shall exceed 1 mm.

NOTE — The position of the junction of the reference thermocouple in the upper (contact) face of the bottom piece shall be 5 mm from the 230 mm edge and 10 mm or less from the bottom edge.

7 Procedure

7.1 Assemble the test piece (or test pieces if two or more tests are being conducted in parallel). In the case of a two-section test piece, place the measuring crosspiece (5.4) and the reference thermocouple (see 5.5) between the sections i.e. in the plane of the hot wire (see figure 2). In the case of a three-section test piece, place the crosspiece, with the hot wire, between the middle and bottom sections and the reference thermocouple between the top and middle sections (see figure 1).

7.2 With a test piece of a dense refractory, the crosspiece and the reference thermocouple shall be cemented in the grooves cut for them (see 6.4) using a cement made from a finely ground quantity of the test material mixed with a small amount of a suitable binder (for example 2 % dextrin and water).

The cement shall be dried before the test is commenced.

7.3 If the test is being performed on powdered or granular material, fill the lower or the bottom container (5.7) with the test material level with its top, and place on it the hot wire and crosspiece and, if a two-section test piece is being used, the reference thermocouple. Place an open container on top of the first and fill it with the test material; if a two-section test piece is being used, this completes the test piece. If a three-section test piece is being used, place the reference couple in position over the middle section and place and fill the upper section in a similar manner. Determine the apparent bulk density of the test material in the poured, untamped state.

7.4 Place the test piece or test pieces in the furnace (5.1), resting each piece (to ensure uniform heating) on two supports, of the same material as the test piece, having dimensions 125 mm × 10 mm × 20 mm, resting on a 125 mm × 10 mm face, and placed parallel to the 100 mm (or 114 mm) faces of the test piece and about 20 mm from those faces.

7.5 Connect the measuring circuits of each test piece to the measuring apparatus (see 5.5 and 5.6).

7.6 With the hot-wire circuit open, raise the temperature of the furnace to the test temperature (the lowest of the test temperature if the test is being performed at more than one temperature) at not more than 10 °C/min.

NOTE — Heating rates should be low enough to ensure that there is no risk of thermal shock damage.

7.7 With the power supply (5.3) connected to a dummy resistance equivalent in value to that of the hot wire, regulate the power input to a value that (from preliminary tests) is known to produce in the hot wire a temperature increase of not more than 100 °C in 15 min.

7.8 When the furnace reaches the test temperature, verify that the temperature in the region occupied by the test pieces is uniform and constant. The variation between the temperatures indicated by the thermocouples connected back to back (the thermocouple welded to the hot wire and the reference thermocouple) shall not be more than 0,05 °C during the time of measurement.

7.9 When the conditions of 7.8 are satisfied, close the hot-wire heating circuit and from that moment measure the elapsed time and make a continuous record of the temperature of the hot-wire, if not using an automatically controlled supply. Measure and record the voltage drop across the hot wire and the current in it immediately after switching on the heating circuit and at intervals of 2 min, including the moments when the elapsed time reaches the values t_1 and t_2 selected when calculating the result (usually 2 min and 10 min).

7.10 After the measuring time, normally 10 to 15 min, switch off the heating circuit current and allow the hot wire and test piece to come to a temperature equilibrium.

7.11 Verify the uniformity and constancy of the temperature in accordance with 7.8. Repeat the procedures of 7.9 and 7.10, so obtaining a further measurement of the rate of rise of the temperature of the hot wire under the same conditions.

7.12 Repeat the procedure described in 7.11, so obtaining a third measurement of the rate of rise of the temperature of the hot wire under the same conditions.

7.13 If the test is also being performed at one or more higher test temperatures, raise the temperature of the furnace to the next higher test temperature at not more than 10 °C/min. Perform again the tests specified in 7.7 to 7.12, so obtaining three measurements of the rate of rise of temperature of the hot wire at that test temperature.

7.14 Repeat the procedure specified in 7.13 until three measurements of the rate of rise of temperature of the hot wire have been obtained at each of the required test temperatures.

8 Assessment of results

8.1 If the current in the hot wire has varied by more than 2 % during the test, the results shall be disregarded and the test shall be carried out again with a current of a smaller value.

8.2 The rise of temperature of the hot wire with respect to time follows a logarithmic law. Recording the temperature rise as a function of time on semi-logarithmic paper should therefore produce a straight-line graph. If this is not the case, either the material does not fulfil the conditions necessary for this test and the results have no significance, or an operating error has been made and the test shall be repeated.

8.3 If the plot of temperature against time (as in 8.2) is non-linear at the lower end, this may be due to the influence of the material surrounding the hot wire. A valid result may possibly be obtained by choosing another value for t_1.

8.4 If the plot of temperature against time (as in 8.2) is non-linear at the upper end, this may be due to the diffusivity of the material under test having too high a value. A valid result may possibly be obtained by choosing another value for t_2.

9 Expression of results

9.1 Method of calculation

The thermal conductivity of the material at each test temperature, λ, is given, in watts per metre kelvin, by the equation

$$\lambda = \frac{I^2 R}{4\pi} \times \frac{\ln (t_2/t_1)}{\Delta\theta_2 - \Delta\theta_1}$$

or

$$\frac{VI}{4\pi} \times \frac{\ln (t_2/t_1)}{\Delta\theta_2 - \Delta\theta_1}$$

where

I is the heating current, in amperes;

V is the voltage drop per unit length of the hot wire, in volts per metre;

R is the electrical resistance per unit length of the hot wire at the test temperature, in ohms per metre;

t_1 and t_2 are elapsed times, in minutes, after closing the heating circuit;

$\Delta\theta_1$ and $\Delta\theta_2$ are the increases in temperature, in kelvins, of the hot wire, after the closing of the heating circuit, at times t_1 and t_2.

9.2 Repeatability

On the basis of the results obtained with the different sets of requirements known to be in use, the repeatability of the method, assessed by its coefficient of variation, is of the order of 8 %.

10 Test report

The test report shall include the following information:

a) the testing establishment;

b) the date of the test;

c) a reference to this International Standard, i.e. "Determination of thermal conductivity by the hot-wire method in accordance with ISO 8894-1";

d) the material tested (manufacturer, product, type, batch number, etc.);

e) in the case of bonded bricks or unfired bricks, the pretreatment given to the test pieces (see the note in 1.3).

f) in the case of unshaped or loose materials (powders or granular materials), the method of preparing the test pieces and the apparent bulk density of the material (i.e. its bulk density in the poured untamped state);

g) the furnace atmosphere;

h) the test temperature or temperatures, and for each of them the three individual values and the mean value of thermal conductivity determined by the test.

NOTE — The individual values are used to establish the mean value, and the mean value is used in further statistical analysis, for example in accordance with ISO 5022.