SLOVENSKI STANDARD
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Metode za preskušanje gostih oblikovanih ognjevzdržnih izdelkov - 14. del: Ugotavljanje toplotne prevodnosti z metodo vroče žice (navzkrižnih žic)

Methods of testing dense shaped refractory products - Part 14: Determination of thermal conductivity by the hot-wire (cross-array) method

Prüfverfahren für dichte geformte feuerfeste Erzeugnisse -Teil 14: Bestimmung der Wärmeleitfähigkeit nach dem Heißdraht-(Kreuz-) Verfahren

Méthodes d'essai pour produits réfractaires façonnés denses - Partie 14: Détermination de la conductivité thermique par la méthode du fil chaud (croisillon)

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This European Standard was approved by CEN on 23 March 1998.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Foreword

This European Standard has been prepared by Technical Committee CEN/TC 187 "Refractory products and materials", the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 1998, and conflicting national standards shall be withdrawn at the latest by October 1998.

It is closely based on the corresponding International Standard, ISO 8894-1 "Refractory materials - Determination of thermal conductivity Part 1 Hot-wire method (cross-array)", published by the International Organization for Standardization (ISO).

The determination of thermal conductivity by the hot-wire (parallel) method is given in EN 993-15.

EN 993 'Methods of testing dense shaped refractory products' consists of 18 Parts:

Part 1 : Determination of bulk density, apparent porosity and true porosity
Part 2 : Determination of true density
Part 3 : Test methods for carbon-containing refractories
Part 4 : Determination of permeability to gases
Part 5 : Determination of cold crushing strength
Part 6 : Determination of modulus rupture, ambient temperatures
Part 7 : Determination of modulus rupture, elevated temperatures
Part 8 : Determination of refractoriness-under-load
Part 9 : Determination of creep in compression
Part 10 : Determination of permanent change in dimensions on heating
Part 11 : Determination of resistance to thermal shock (ENV)
Part 12 : Determination of pyrometric cone equivalent
Part 13 : Specification for pyrometric cones
Part 14 : Determination of thermal conductivity by the hot wire (cross-array) method
Part 15 : Determination of thermal conductivity by the hot wire (parallel) method
Part 16 : Determination of resistance to acids
Part 17 : Determination of bulk density of granular material (mercury method)
Part 18 : Determination of bulk density of granular material (water method)

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This Part of EN 993 specifies a hot-wire (cross-array) method for the determination of thermal conductivity of refractory products and materials. It is applicable to dense and insulating shaped products and to powdered or granular materials (see 7.3), for thermal conductivities of less than 1,5 W/m·K. Electrically conducting materials cannot be measured.

NOTE 1 : The thermal conductivity of bonded bricks may be affected by the appreciable amount of water that is retained after hardening or 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 the measurement temperature, as a preliminary to carrying out the test, are details that are outside the scope of this Part of EN 993 and should be agreed between the parties concerned.

NOTE 2 : In general it is difficult to make measurements on anisotropic materials. The use of this method for such materials should also be agreed between the parties concerned.

2 Normative references

This European Standard incorporates by dated or undated references, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN 993-15	Methods of test for dense shaped refractory products - Determination of thermal conductivity by the hot wire (cross-array) method
ISO 31-4	Specification for quantities, units and symbols. Heat

3 Definitions

For the purposes of this standard, the following definitions, in accordance with ISO 31, apply.

3.1 thermal conductivity, λ: density of heat flow rate divided by the temperature gradient, in units of watt per metre kelvin (W/m·K).

3.2 thermal diffusivity, α: $\alpha = \frac{\lambda}{\rho . C_p}$,

where
- λ is the thermal conductivity

- ρ is the bulk density
- C_p is the specific heat capacity at constant pressure.

Thermal diffusivity is expressed in units of square metre per second ($m^2 \cdot s^{-1}$).

3.3 power, *P*: Rate of energy transfer, in units of watts (W).

4 Principle

The test pieces are heated in a furnace to a specified temperature and maintained at that temperature. Further local heating is provided by a linear electrical conductor (the hot wire) that is embedded in the test piece and carries an electrical current of known power that is constant in time and along the length of the test piece.

The thermal conductivity is calculated 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 material from which the test pieces are made.

5 Apparatus

5.1 Furnace, electrically heated, capable of taking one or more test assemblies (see 6.1) up to a maximum temperature of 1250 °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 measured on the outside of the test assembly during a test (of duration about 15 min) shall not vary by more than ± 0,5 °C, and shall be known with an accuracy of ± 10 °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 actual length being known to within ± 0,5 mm. The value of the electrical reistance of the wire shall be known.

NOTE : A hot wire made of base metal can be used at temperatures below 1000 °C.

5.3 Power supply, to the hot wire (see 5.2), which shall be stabilized a.c. or d.c. and shall not vary in power by more than 2 % during the period of measurement. A constant power supply is preferred.

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 circuit. 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 shall be 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 Digital multimeter, for measuring the current in the hot wire and the voltage drop between the ends, and capable of measuring both to an accuracy of at least ± 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 of the resistance of the hot wire with temperature (see 7.9).

5.6.2 Data acquisition system, consisting of a temperature-time registration device with a sensitivity of at least 2 μV/cm or 0,05 μV/Digit, or a temperature measurement accuracy of 0,05 °C with a time resolution better than 0,5 s.

5.7 Containers (for use if the test is performed on powdered or granular material), having internal dimensions equal to those of the solid test assembly specified in clause 6, so that the test assembly shall consist of either two or three sections as specified in 6.1. 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.

NOTE : Containers should be of a material that will not react with the test piece at the temperature and should not be electrically conducting.

6 Test pieces

6.1 Dimensions

Each test assembly shall consist of two or three identical test pieces, not less than 200 mm x 100 mm x 50 mm in size.

NOTE : It is recommended that the size of each test piece be 230 mm x 114 mm x 64 mm or 230 mm x 114 mm x 76 mm. Standard-size bricks should then be used as the pieces forming the test assembly, subject to the requirements of 6.2.

6.2 Surface flatness

The surfaces of the two test pieces forming the test assembly which are in contact with each other 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.

6.3 Grooves in dense materials

In dense materials, when a two-section test piece is used, two straight grooves for the measuring crosspiece (see 5.4) and a V-groove for the reference thermocouple (see 5.5) shall be machined in the upper (contact) face of the lower section (see figure 1). 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 should be 5 mm from the 230 mm edge and 10 mm or less from the 114 mm 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 (see 5.4) and the reference thermocouple (see 5.5) between the sections, in the plane of the hot wire (see figure 1). 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 2).

7.2 With a test piece of a dense refractory, cement the crosspiece and the reference thermocouple in the grooves cut for them (see 6.3) 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).

Dry the cement 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 (see 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.

NOTE : The container can be filled by vibration or by pressing to give a specific bulk density, where a figure has been agreed upon.