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Refractory materials - Determination of thermal conductivity (calorimeter)

耐火材料 导热系数试验方法 (水流量平板法)

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Refractory materials - Determination of thermal conductivity (calorimeter)

1 Scope

This Standard specifies the definition, principle, equipment, specimens, test procedures, test error, etc. of test method for refractory material thermal conductivity (calorimeter).

This Standard applies to the determination of the thermal conductivity of refractory materials with a hot surface temperature of $200^{\circ}\text{C}\sim1300^{\circ}\text{C}$ and a thermal conductivity of $0.03~\text{W/(m\cdot K)}\sim2.00~\text{W/(m\cdot K)}$.

2 Normative references

The provisions in following documents become the provisions of this Standard through reference in this Standard. For dated references, the subsequent amendments (excluding corrigendum) or revisions do not apply to this Standard, however, parties who reach an agreement based on this Standard are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

GB/T 8170, Rules of rounding off for numerical values and expression and judgement of limiting values

3 Definitions

Thermal conductivity refers to the heat transferred through the unit area of the material along the heat flow direction under the unit temperature gradient per unit time. It can be represented as:

$$\lambda = q/(\mathrm{d}T/\mathrm{d}x) \tag{1}$$

Where,

 λ - the thermal conductivity, in watts per meter Kelvin (W/(m·K));

q - the heat flux density per unit time, in watts per square meter (W/m^2) ;

dT/dx - a temperature gradient, in Kelvin per meter (K/m).

surface; 8. Terminal; 9. Insulating porcelain tube; 10. Protective cover; 11. High aluminum fiber; 12. Supporting block; 13. Specimen; 14. Backing plate; 15. Glass fiber cloth; 16. Calorimeter

5.2 Calorimeter system

- **5.2.1** The central calorimeter, the first protective calorimeter and the second protective calorimeter are all made of red copper material with small specific heat and good thermal conductivity. Make sure that the three calorimeters are on the same level.
- **5.2.2** The central calorimeter is a double-circuit water channel to ensure that the temperature of the calorimeter is uniform.
- **5.2.3** The temperature rise thermocouple stack is made of 10 pairs of Φ 3 mm copperconstantant hermocouple wires, which are used to measure the temperature rise of the water flowing in and out of the central calorimeter.
- **5.2.4** The temperature difference zeroing thermocouple stack is made of 8 pairs of Φ 0.3 mm copper-constantan thermocouple wires, which are used to measure the temperature difference between the central calorimeter and the first protection calorimeter.

5.3 Water supply system

The water flow rate of the central calorimeter should be adjustable within the range of $30 \text{ g/min} \sim 120 \text{ g/min}$.

The constant pressure water tank shall have upper water, lower water and overflow devices to maintain stable water pressure during the test. Make sure that the water temperature fluctuation is not greater than 0.6°C during the test. The constant pressure water tank is installed about 2.5 m above the ground.

5.4 Fixing ring for measuring bulk material

The ring made of refractory material has an inner diameter of (180 ± 2) mm and an outer diameter of (210 ± 2) mm.

5.5 Backing plate

5.6 Other appliances

- **5.6.1** Thermocouple: Φ 0.5 mm S-type lead-rhodium 10-platinum thermocouple is used to control the temperature of the cold and hot surfaces of the specimen and the temperature of the heating furnace. Thermocouples should be calibrated periodically.
- **5.6.2** Electrical signal measuring device: use UJ33a DC potentiometer or electrical signal measuring instruments above 0.05 level.
- **5.6.3** Stopwatch: the resolution is 0.1 s.
- **5.6.4** Vernier caliper: the division value is 0.02 mm.

7.3 Furnace loading

- **7.3.1** Put the prepared glass fiber cloth with a diameter of 220 mm and the backing plate of the required material on the calorimeter in sequence. Place the ends of the thermocouples measuring the temperature of the cold side at the center of the backing plate.
- **7.3.2** Place the specimen on the backing plate (on the cold side thermocouple). Use hand to gently press. Make a minimum gap between the backing plate and the specimen.
- **7.3.3** Place support blocks made of lightweight material on the edge of the specimen (one every 120°). Then fill the gaps around the specimen with high alumina fiber cotton.
- **7.3.4** Place the hot junction of the thermocouple measuring the temperature of the hot surface at the center of the hot surface of the specimen.
- **7.3.5** Place the vapor chamber on the support block so that the vapor chamber is parallel to the specimen. The spacing is $10 \text{ mm} \sim 15 \text{ mm}$. Cover tightly with fiber felt (blanket) around the vapor chamber. The calorimeter, pad, backing plate, specimen and vapor chamber should be coaxial.

Cover the furnace cover. Make it have no gap with the lower part of the furnace.

7.4 Heating

Heat according to one of the following provisions:

- a) Generally, the specimen is heated from room temperature to the test temperature at a temperature not greater than 10°C/min. Keep temperature constant at the test temperature for 50 min.
- b) For silicon products, heat up at 5°C/min from room temperature to test temperature. Keep temperature constant at the test temperature for 50 min.
- c) For monolithic refractory materials, from room temperature to test temperature, the temperature should be increased by no more than 10°C/min. Keep temperature constant at the test temperature for 120 min.
- d) Heat up according to the process requirements of the product.

7.5 Measurement

- **7.5.1** Regulate the water flow to the central calorimeter. The flow rate is determined according to the material of the specimen, which is generally controlled within the range of $30 \text{ g/min} \sim 120 \text{ g/min}$.
- **7.5.2** Adjust the water flow of the first protection calorimeter so that the temperature difference between the central calorimeter and the first protection calorimeter is zero.

Allow fluctuation ± 0.005 mV.

- **7.5.3** Measure the potential of hot surface thermocouple and cold surface thermocouple.
- **7.5.4** Measure the water temperature rise, i.e.: the potential of 10 pairs of thermocouples.
- **7.5.5** Measure the water flow in central calorimeters. Each test temperature point is measured three times. Measure once every 10 min. Then calculate the average value. The deviation of each measured value from the average value is not more than 10%, otherwise it should be re-measured.

8 Calculation

8.1 Calculate the thermal conductivity according to formula (4):

$$\lambda = k \cdot \Delta m v \cdot w \cdot \delta / (t_1 - t_2) \tag{4}$$

Where,

 λ - the thermal conductivity, in watts per meter Kelvin (W/(m·K));

k - the constant, in joules per gram millivolt square meter $(J/(g \cdot mV \cdot m^2);$

 Δmv - the electromotive force difference of the water temperature rise of the central calorimeter, in millivolts (mV);

- w the water flow of the central calorimeter, in grams per second (g/s);
- δ the thickness of the specimen, in meters (m);
- t₁ the temperature of the hot surface of the specimen, in degrees (°C);
- t₂ the temperature of the cold surface of the specimen, in degrees (°C).
- **8.2** Calculated results are rounded to three decimal places according to GB/T 8170.

9 Test report

The test report shall at least contain the followings:

- a) Specimen number;
- b) Specimen name;
- c) Thickness of the specimen;

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