AN APPARATUS FOR MEASURING THE THERMAL CONDUCTIVITY AND DIFFUSIVITY OF SOLID MATERIALS

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An apparatus is described for measuring the thermal conductivity and diffusivity on small specimens of solid materials; also the results are shown which have been obtained for refractive high-alumina concrete by such measurements.

When performing measurements with this apparatus, one applies either the method of two plates for determining the thermal conductivity [1, 2] or the method of the dynamic a-calorimeter [3]. The first method is static and requires 3-4 h for obtaining one value of thermal conductivity at a given temperature. The second method is based on the relations characterizing a regular heating mode and yields the thermal diffusivity over the entire 100-500°C temperature range in a single test.

The thermal conductivity of test materials should not exceed 5 W/m · °C. The specimens have the shape of disks 20 mm in diameter and 5-7 mm thick. The thermal conductivity is measured over the range from room temperature to 800°C. Provisions are made for measurements in air, in an inert atmosphere, or in vacuum.

The construction of this apparatus is shown schematically in Fig. 1. Two identical specimens 5 of the test material are placed on both sides of a flat electric heater 4 at the center, a heater made of nichrome wire, and are pressed tightly between metal blocks 6 by means of a spring 10. Into blocks 6 are built in end heaters 3 and water coolers 9. On the guard cylinder 8 made of stainless steel are placed three lateral heaters 7. The space between cylinder 8 and housing 2 of the apparatus is filled with thermal insulation (zirconia). The inner cavity is sealed hermetically through gaskets 1. The thermocouple and heater leads are brought out through a sleeve 11 with double gaskets. The outer thermal insulation 12 consists of baked asbestos. For convenient assembly, the apparatus can be rotated about its horizontal axis on the stand 13.

The temperature field in the apparatus is measured with eleven Chromel–Alumel thermocouples, five of which are installed on the guard cylinder 8, two in the metal blocks 6, and four in the specimens 5. The thermocouples are made of 0.2 mm (diameter) wire pulled through twin ceramic sleeving 1.3 mm in diameter. For the installation of the thermocouples in specimens one cuts grooves in the specimen surfaces approximately 1.5 mm deep and, after the thermocouples have been embedded and the distance between thermocouple beads has been measured, these grooves are filled with high-temperature paste.

The thermal emf of the thermocouples is measured with a model PMS-48 or a model R375 potentiometer. The central heater 4 is supplied from a rectified and stabilized voltage source; its electric power is measured with a class 0.2-0.5 wattmeter. The end heaters 3 and the lateral heaters 7 operate on alternating current; their power is regulated through laboratory autotransformers. The active space in the apparatus is cleared of air by means of a model RVN-20 vacuum pump and then filled with inert gas. For a measurement of thermal conductivity the test is performed with the powers of the central heater 4 and of the middle lateral heater 7 matched so as to produce a 20-40°C temperature drop across the specimens, while bringing the temperature at each specimen section close to the temperature at the opposite surface

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of the guard cylinder 8. For low-temperature tests one turns on the water coolers 9, for high-temperature tests one turns on the end heaters 3. The two outer lateral heaters 7 are turned on when it is necessary to level the temperature distribution in the guard cylinder 8. After steady state has been reached, when $\partial t/\partial \tau < 0.2$ °C/min, all thermocouples and the wattmeter of the central heater are read. The thermal conductivity is then calculated by the formula

$$\lambda = \frac{Q}{F\left(\frac{\Delta t_1}{\delta_1} + \frac{\Delta t_2}{\delta_2}\right)}. \tag{1}$$

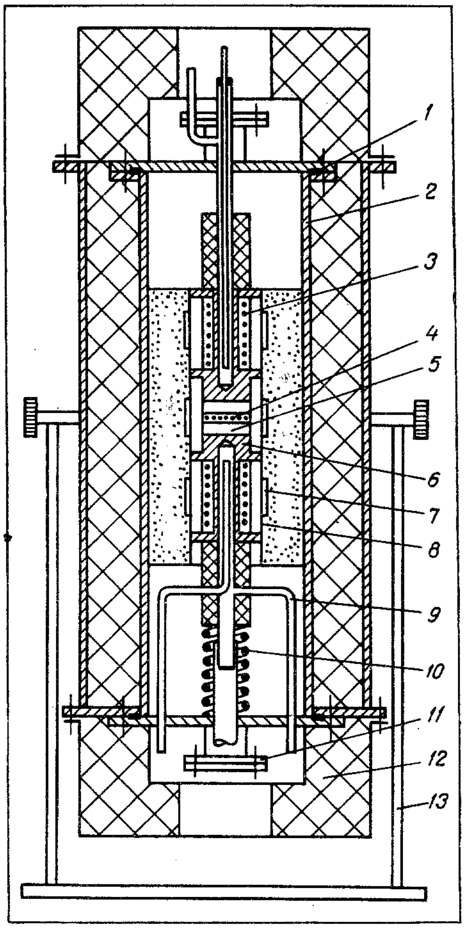

Fig. 1. Schematic diagram of test apparatus.

The repeatability of thermal conductivity measurements depends essentially on how precisely adiabatic the conditions are at the lateral surfaces of the specimens and how accurately the temperature drops across the specimens are measured. Since thermocouples mounted in the specimens often fall out of adjustment, they are calibrated directly in the apparatus. For this purpose one requires isothermal conditions, which prevail when the readings of calibrated thermocouples in blocks 6 and in guard cylinder 8 are the same. This is achieved by regulating the end heaters 3 and the lateral heaters 7 with the central heater 4 turned off.

In addition, the results may become distorted by a nonuniform thermal resistance over the contact surfaces. In order to avoid this, the end surfaces of specimens must have a fine finish and the applied pressure must not be less than 5 kg/cm^2.

For thermal diffusivity measurements one uses the same specimens, but the central heater 4 is now removed. The end heaters 3 and the two outer lateral heaters 7 are set at a certain constant power level so as to produce, when nominal steady state is reached, a maximum apparatus temperature 2-3 times higher than the maximum measurement temperature. The mean-over-the-height temperature of specimens during heating should be equal to the mean temperature at the opposite surface of guard cylinder 8. During the heatup one reads all thermocouples. The thermal diffusivity is then calculated by the following formula:

$$a = \frac{\partial t(d_1, \tau)}{\partial \tau} \cdot \frac{d_2^2}{\Sigma \Delta t} \cdot \frac{2(\cos \nu d_1 - \cos \nu d_2)}{(\nu d_2)^2 (\cos \nu d_1 + b \sin \nu d_1)}. \tag{2}$$

The derivation of (2) is analogous to the derivation of the formula in [3]. When $d_1 = 0$ and $d_2 = d$, both expressions become identical.

On the basis of the theoretical principles of this method, the thermal diffusivity can be determined over a temperature range within which specimens are heating up exponentially (regular mode). This stipulation limits the temperature range of measurements from below (initial heating period) as well as from above (approach to steady state) and, with thermocouple and heater characteristics also taken into account, the temperature range is thus cut to approximately 100-500°C. In order to yield reliable thermal diffusivity data, the thermal resistance of the contact between specimen and block surfaces must be lower than that of the specimens. Otherwise, the thermal flux distribution may become nonuniform over the specimen section or the upper and the lower specimen will heat up very unequally.

This apparatus has been proved out in measurements involving many engineering materials. As an example, we present here data pertaining to the thermal conductivity and diffusivity of unbaked refractive high-alumina concrete. The composition of the material was: 80% Al_2O_3 + 20% high-alumina cement (79% $CaO \cdot 2Al_2O_3$, 10% $2CaO \cdot Al_2O_3$, 1% $MgO \cdot Al_2O_3$, 9% Al_2O_3). The granulometric content of the alumina was: 50% corundum No. 80 (800-1000 μ), 30% corundum No. 50 (500-630 μ), and 20% corundum No. 10 (100-125 μ). The density of specimens ranged from 2.80 to 2.85 kg/cm^3.

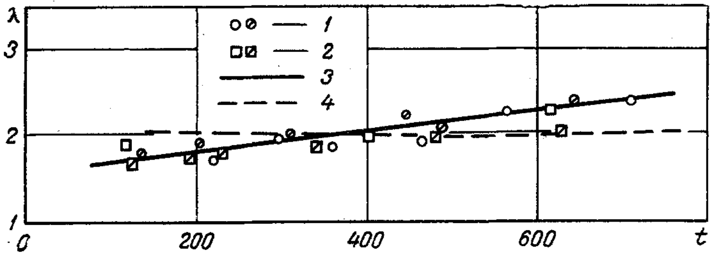

Fig. 2. Thermal conductivity of refractive high-alumina concrete: 1) first and second test series; 2) first and second test series by the method of one plate; 3) averaging straight line; 4) data from [4]. Thermal conductivity λ (W/m · °C), temperature t (°C).

The thermal conductivity of this refractive concrete was measured in air at temperatures varying up to 700°C. The thermal resistance of the contacts between specimens and blocks was reduced by means of thin layers of paste (alumina powder dispersed in liquid glass) on the end surfaces. After the first series of tests, the specimens were removed from the apparatus and then reinstalled with different thermocouples for a second series of tests. The results of these measurements are shown in Fig. 2. They have been averaged with a straight line according to the method of least squares. The relative random error of the thermal conductivity measurement is here ±3% at a 95% confidence level [7]. This error is due to differences in the calibration and the installation of thermocouples in the specimens, also due to deviations from adiabaticity during tests. The systematic error of the thermal conductivity measurement is estimated within 5-6%, based on relative instrument inaccuracies: Q(0.2-0.5%), F(2%), Δt(1-2%), and δ(1.5-2.0%). Thus, the total error of the thermal conductivity measurement is 8-9%. The maximum deviation of test points from the averaging line does not exceed 9%.

In Fig. 2 are also shown test points obtained with the same specimens on another apparatus by the method of one plate. By this method, too, two test series were performed. The data obtained by both methods on two different test stands respectively are in close agreement within the scatter of test points.

The repeatability of results in each of the four test series indicates that heating refractive high-alumina concrete to 600-700°C does not produce any significant structural changes in the material as a result, for instance, of the removal of crystallization water. This is confirmed by the data in [4], where the thermal conductivity of unbaked and baked (at 1200°C) refractive high-alumina concrete specimens was measured by the method of radial heat flow. Up to about 800°C a considerable difference was observed in [4] between the thermal conductivity of baked and unbaked material, due to the presence of crystallization water in the latter. A comparison of our results with the data on unbaked refractive high-alumina concrete in [4] shows that the respective test values agree at temperatures from 300 to 400°C but differ by 17-18% at the top temperature. Such a discrepancy is, apparently, due to differences between test specimens.

The thermal diffusivity of refractive concrete was measured on the same specimens. In preliminary tests the heater powers were adjusted so as to heat up the specimens and the guard cylinder 8 at the same rate. This ensured an only negligible leakage of heat through the lateral specimen surfaces. Two subsequent basic tests yielded data necessary for the determination of thermal diffusivity. In the first of these tests the end heaters 3 were turned on to full power, in the second test they were turned down to an approximately 30% power level. The heating rate and the temperature drops in the specimens were calculated from thermograms (Fig. 3). The range of valid measurements, corresponding to a linear $\partial t/\partial \tau = f(t)$ relation and an exponential $t = f(\tau)$ relation, was established on the basis of the trend of the heating rate versus temperature curve. In the first test this range was 150-450°C; in the second test it was 100-250°C. Within these ranges, with smoothed out values of $\partial t/\partial \tau$ and $\Sigma \Delta t$ in Fig. 3, the values of thermal diffusivity were calculated for refractive high-alumina concrete as shown in Fig. 4. The correction factor (the third factor in Eq. (2)) was very close to unity.

The random error of the thermal diffusivity measurement is approximately 1% at a 95% confidence level. The systematic error due to instrument inaccuracy is estimated at 5-7%. The total error is 6-8%.

From these values of thermal conductivity and diffusivity, the true specific heat of refractive concrete was estimated according to $c = \lambda/a\gamma$. Within the 150-450°C range it increases from 0.88 to 1.24 kJ/kg · °C. A

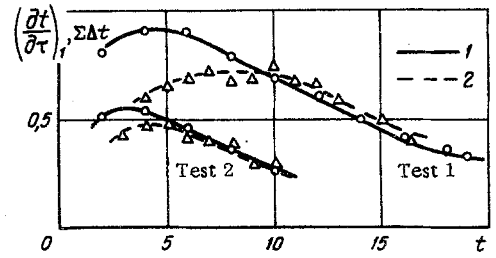

Fig. 3

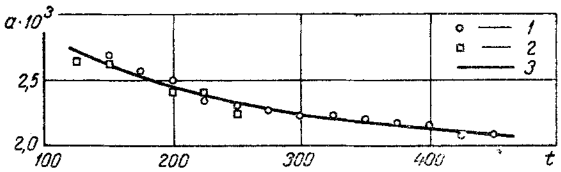

Fig. 4

Fig. 3. Heating rate and temperature drop in specimens, as functions of the temperature: 1) $(\partial t/\partial \tau)_1$, mV/min; 2) $\Sigma\Delta t$, mV. Temperature t (mV).

Fig. 4. Thermal diffusivity of refractive high-alumina concrete: 1) first test; 2) second test; 3) averaging straight line. Thermal diffusivity a (m^2/sec), temperature t (°C).

comparison with the specific heat determined on the basis of data in [5, 6] for refractive high-alumina concrete (chemical composition: 84% Al_2O_3 + 16% $CaO \cdot 2Al_2O_3$) shows a mean difference of approximately 4.2% and a maximum difference of 8.3% within that temperature range.

NOTATION

λ	is the thermal conductivity at the mean temperature of specimens, W/m · °C;
Q	is the power of the central heater, W;
F	is the cross section area of a specimen, m^2;
$\Delta t_{1,2}$	is the temperature drop across the specimens, °C;
δ_1, δ_2	is the difference in heights between the thermocouple beads, center-to-center, in the first and in the second specimen respectively, m;
t	is the temperature, °C;
τ	is the time coordinate, min;
$d_1 = (d_{1u} + d_{1l})/2$	is the mean distance between specimen contact plane and nearest thermocouple beads, for the upper and lower specimen, m;
$d_2 = (d_{2u} + d_{2l})/2$	is the mean distance between specimen contact plane and farthest thermocouple beads, for the upper and lower specimen, m;
$dt(d_1, \tau)/d\tau$	is the rate of temperature rise at section d_1 of the specimen at time τ, °C/h;
$\Sigma\Delta t = \Delta t_1 + \Delta t_2$	is the sum of temperature drops in the specimens at time τ, °C;
m	is the heating rate, h^{-1};
a	is the thermal diffusivity of specimens, referred to their mean temperature, m^2/h;
$\nu = \sqrt{m/a}$, m^{-1}	
$b = \lvert(\Delta t_u - \Delta t_l)/\Delta t_u\rvert$	is the heating nonuniformity factor.

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