

AN INVESTIGATION OF THE THERMAL DIFFUSIVITY AND THERMAL CONDUCTIVITY OF INSULATING POWDERS AT ATMOSPHERIC PRESSURE AND IN VACUUM BY VARIOUS METHODS

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This paper gives the results of measurements of the thermal diffusivity and thermal conductivity of an aerogel, perlite powder, and white soot by the steady-state method, the continuous heating method, and the method of the regular regime of the first kind. The experimental data agree well with theory.

A determination of the temperature dependence of the thermal conductivity of insulating materials can be used to assess the role of different factors in the transfer of heat through insulation and to develop methods of reducing such transfer.

Determinations by the steady-state heat flux method require several experiments with different temperatures on one of the boundary walls.

The method of continuous variation of the temperature [1-3] can be used to find the temperature dependence of the thermal diffusivity or thermal conductivity in one experiment. For the particular case of an infinitely long cylinder the formulas have the form:

$$a = \frac{R^2}{4\Delta T} \frac{d}{d\tau} \left( T_0 + \frac{1}{4} \Delta T \right) - \frac{5}{2304} \frac{R^4}{a\Delta T} \frac{d^2 \Delta T}{d\tau^2}, \quad (1)$$

$$\lambda = \frac{1}{2\Delta T} \frac{dT}{d\tau} \left( R_c^2 c_c \gamma_c \ln \frac{R}{R_c} + c\gamma \frac{R^2 - R_c^2}{2} - R_c^2 c\gamma \ln \frac{R}{R_c} \right). \quad (2)$$

The error due to the finite value of the ratio L/R is negligible if this ratio is not less than 4-5. The continuous heating method was used in [3] for measurements of heat-insulating materials at temperatures of 100-600° C.

We consider the possibility of using the method of the regular heat regime of the first kind to find the temperature dependence of  $a$  and  $\lambda$ . The equations obtained for this method are derived on the assumption of constancy of  $\lambda$ ,  $c$ , and  $a$ . In particular, for a cylinder,

$$a = \frac{m}{\left( \frac{2.4048}{R} \right)^2 + \left( \frac{\pi}{L} \right)^2}. \quad (3)$$

If  $\lambda$  and  $c$  vary with temperature in such a way that  $a$  remains constant, the use of these equations will not lead to significant errors when the values of  $(1/\lambda) \times (d\lambda/dT)$  and  $(1/c)(dc/dT)$  are relatively small. If  $a$  also varies, the rate of cooling will change continuously during the experiment. We can assume that in a first approximation the rate of cooling and the

thermal diffusivity will still be proportional, in accordance with (3).

In this work we measured  $a$  and  $\lambda$  of several insulating powders in the range 78-300° K by the steady-state heat flux method, the continuous heating method, and the method of the regular heat regime of the first kind. In the steady-state method  $\lambda$  was measured by a sphere on the apparatus described in [4]. For measurements of  $a$  and  $\lambda$  by the unsteady-state methods we constructed two calorimeters, consisting of copper cylinders with an internal diameter of 35 mm and a length of 190 mm. On the axis of the  $a$ -calorimeter there was a manganin-constantan thermocouple (wire diameter 0.05 mm); with the junction at the central point of the cylinder. The junction of another thermocouple was soldered to the copper surface. An electric heater for regulating the heating rate was mounted on the outer surface. The  $\lambda$ -calorimeter had the same shell dimensions. Suspended in the center of it was a core—an aluminum rod 5 mm in diameter and 100 mm long.

The calorimeter was mounted in a bath, insulated with glass wool, and put into a Dewar vessel. The calorimeter was then exhausted to a pressure of less than 1 N/m<sup>2</sup>, the bath and vessel were filled with liquid nitrogen, and an EPP-09 potentiometer recorded the temperature in the center of the sample as it cooled. From the results of the measurements we calculated  $m$  and then  $a$  from (3).

When the sample had cooled to 78° K we switched on the heater, evaporated the liquid nitrogen from the bath and heated the calorimeter at such a rate that the difference in the temperatures at the center and on the periphery of the sample did not exceed 20-30°. In most experiments the difference in the emf of the thermocouples was kept constant. The series of experiments was then repeated with the temperature difference kept constant. The rate of heating in the first case gradually decreased with time. In the second case it increased. The mean difference between the results of these experiments did not exceed 10% and the greatest difference was 25%. It should be noted that the differences were of a systematic nature—the values of  $a$  obtained when  $\Delta T$  was kept constant were usually lower and agreed better with the values obtained by the other methods.

We did not measure  $\lambda$  of the powders in a vacuum, since the introduction of the extra mass of the core made the experiment several times longer than in the case of measurement of  $a$ . For instance, heating of evacuated aerogel through 30° took 6 hr. Hence, we

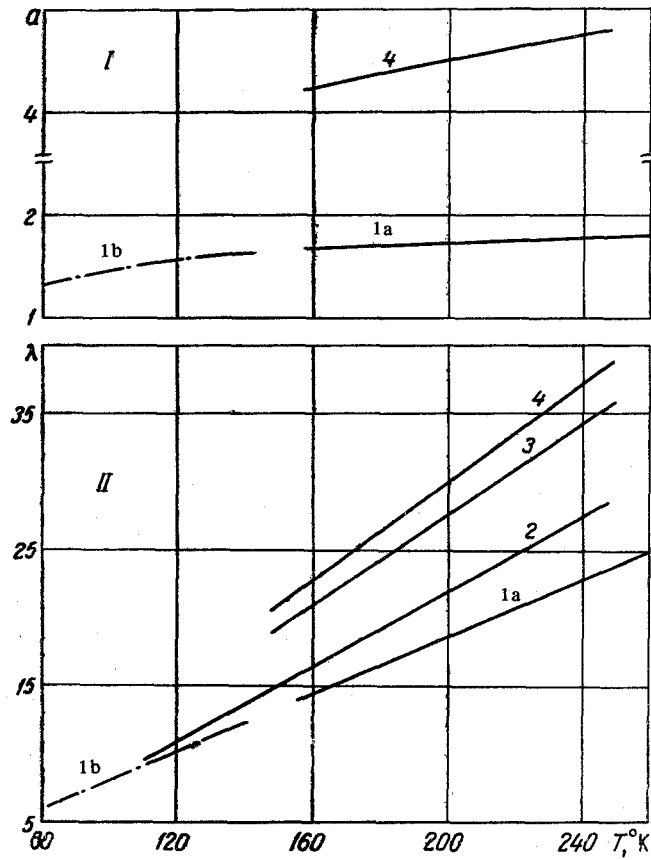


Fig. 1. Temperature dependence of thermo-physical properties of insulating powders at atmospheric pressure: I) thermal diffusivity ( $10^7 \text{ m}^2/\text{sec}$ ); II) apparent thermal conductivity ( $\text{mW}/\text{m} \cdot \text{deg}$ ); 1) BS-280 white soot (*a*—continuous heating method, *b*—regular regime method); 2) aerogel; 3) U-333 white soot; 4) perlite powder.

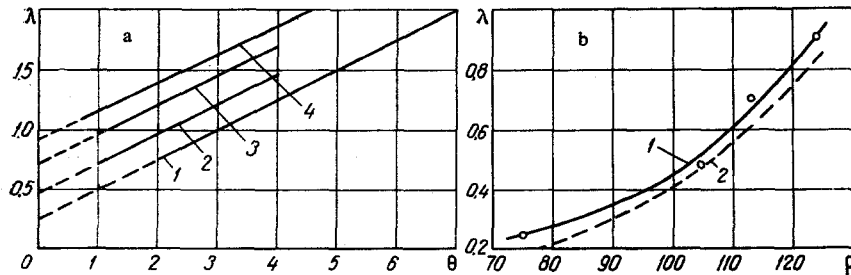


Fig. 2. Apparent thermal conductivity of aerogel ( $\text{mW}/\text{m} \cdot \text{deg}$ ) in vacuum in relation to: a) temperature with different densities,  $\text{kg}/\text{m}^3$  (1—74, 2—105, 3—113, 4—124); b) density [1—experimental data; 2—from equation (6)].  $\Theta \equiv (T_1 + T_2)(T_1^2 + T_2^2) \cdot 10^{-7}$ .

calculated  $\lambda$  from the experimental values of  $a$  by using published values of the specific heat of integral materials of the same chemical composition. The absence of an appreciable difference in the specific heat of dispersed and integral materials of the same composition has been confirmed by recent investigations.

Measurements were made on perlite powder, silicic acid aerogel, and white soot. Perlite powder is finely dispersed, expanded perlite (volcanic glass). The aerogel and white soot are finely powdered silicon dioxide. White soot differs from the aerogel in its greater density and smaller grains, which in both materials consist of aggregates of microparticles with a diameter of about 15 nm (150 Å).

The thermal diffusivity of the insulating powders at atmospheric pressure (Fig. 1, I) in the investigated temperature range did not depend greatly on the temperature. Heat transfer in insulating powders at this pressure is due mainly to conduction by the gas [4]. The temperature dependence of the thermal conductivity is represented by the straight lines (Fig. 1, II).

In measurements by the regular regime and continuous heating methods the initial portions of the relationship are discarded. In the first method this is the high-temperature region and in the second it is the low-temperature region. Hence, the two methods supplement one another and enabled us to cover the whole investigated temperature range (see experimental data for BS-280 white soot in Fig. 1, II, and Fig. 3).

In the experiments to determine the thermophysical properties of evacuated powders the pressure did not exceed 0.4 N/m<sup>2</sup>. Heat transfer by the gas at such pressure becomes negligible and is due almost entirely to conduction by the solid and to radiation. In this case the temperature dependence of the apparent thermal conductivity of the powder can be put in the following form:

$$\lambda = \lambda_{\text{con}} + \lambda_{\text{rad}} = \lambda_{\text{con}} + k(T_1 + T_2)(T_1^2 + T_2^2). \quad (4)$$

The temperature dependence of the refractive index and the thermal conductivity of the solid can be neglected in this case. If the boundary temperatures are close, Eq. (4) becomes

$$\lambda = \lambda_{\text{con}} + 4kT_{\text{av}}^3. \quad (5)$$

When the temperature differences  $((T_1 - T_2)/T_2)$  are relatively low ( $\leq 0.2$ ) the error in using (5) instead of (4) does not exceed 1%. As was shown in [3, 5], the thermal conductivity of several insulating materials in a vacuum conforms with (5).

Figure 2a shows the temperature dependence of the thermal conductivity of aerogels of different compaction in a vacuum. The different compactions were obtained by exhausting the instrument to different pressures after the powder was loaded. The results of the measurements are represented by approximately parallel straight lines. The increase in thermal conductivity with increasing compaction is due to the greater contact heat transfer between the powder particles. A formula for calculating the contact heat transfer was

obtained in [6]; it has the form

$$\lambda_{\text{con}} = \frac{3.12\lambda_s(1 - \epsilon)^{1/3} p^{1/3}}{E^{1/3}} = \frac{3.12\lambda_s \gamma^{1/3} p^{1/3}}{\gamma_s^{1/3} E^{1/3}}. \quad (6)$$

This formula was derived from a model in which the disperse medium was regarded as a system of spherical particles with contact spots on their surface. The

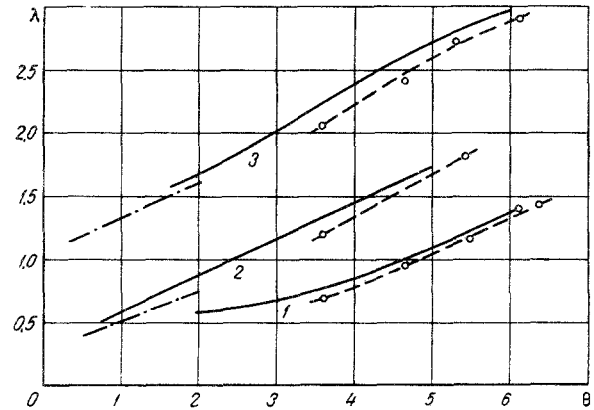


Fig. 3. Temperature dependence of apparent thermal conductivity (mW/m · deg) in vacuum: 1) BS-280 white soot ( $\gamma = 200 \text{ kg/m}^3$ ); 2) perlite powder ( $\gamma = 130 \text{ kg/m}^3$ ); 3) U-333 white soot ( $\gamma = 350 \text{ kg/m}^3$ ). Solid lines—continuous heating method, dashed lines—steady-state method, dot-dash lines—method of regular regime of first kind.  $\Theta \equiv (T_1 + T_2)(T_1^2 + T_2^2) \cdot 10^{-7}$ .

state of the surface (for instance, the presence of an oxide film, microirregularities) which can have a significant effect in the case of relatively large particles of high thermal conductivity, particularly metal particles, should not have an appreciable effect on the thermal resistance of insulating powders for which formula (6) is proposed.

To use the formula we need to know the specific load on the powder. To do this we measured the density of the aerogel under different loads and then calculated the contact thermal conductivity of the aerogel from formula (6).

For the calculations we need to know  $\lambda_s$ , i. e., the thermal conductivity of silicon dioxide in the amorphous state. In a first approximation it can be taken as equal to that of the thermal conductivity of quartz glass: 1.2–1.86 W/m · deg [7]. In the calculations we took  $\lambda_s = 1.6 \text{ W/m} \cdot \text{deg}$ ,  $\gamma_s = 2300 \text{ kg/m}^3$ , and  $E = 5 \cdot 10^{10} \text{ N/m}^2$ . The calculated values are compared in Fig. 2b with the experimental values found by extrapolation of the straight lines in Fig. 2a to the y axis. The agreement is quite satisfactory.

The temperature dependence of the thermal conductivity of perlite in vacuum (Fig. 3) is also represented by a straight line in the coordinates of Eq. (4). Measurements by the three methods gave consistent results. The values of  $\lambda_{\text{con}}$  and  $\lambda_{\text{rad}}$  of perlite and the aerogel are close to one another. For a number of other perlite samples  $\lambda_{\text{rad}}$  was lower, with the result that the apparent thermal conductivity of perlite was lower than that of the aerogel. At the boundary tem-

peratures of 293 and 90° K the mean value was 1.0 mW/m·deg for perlite and 1.4 mW/m·deg for the aerogel. The apparent thermal conductivity of U-333 white soot in the same conditions was approximately twice as high. White soot, as Fig. 3 shows, had higher  $\lambda_{\text{con}}$  and  $\lambda_{\text{rad}}$ . The reasons for this are the greater density (which increases the contact heat transfer), and the small particle size (which reduces the scattering of heat radiation).

The temperature dependence found for the thermal conductivity of BS-280 white soot is of considerable interest. It is represented by a curve which is an almost horizontal straight line at low temperatures. Hence, scattering of radiation by this powder increases rapidly when the temperature is reduced. This explains the relatively low thermal conductivity (0.7 mW/m·deg) at the above-indicated boundary temperatures.

The radiation scattering coefficient of BS-280 soot depends on the phase shift

$$\rho = 2(n-1) \frac{\pi d}{\lambda_w} \quad (7)$$

and reaches a maximum when  $\rho \cong 4$ . The soot grains have a diameter of approximately 10  $\mu$ . For an exploratory calculation of the refractive index of this material in the infrared region we take it as equal to that of quartz and quartz glass at wavelength 10–3.6  $\mu$  [8]. We find from formula (7) that the greatest scattering of heat radiation by particles of BS-280 soot occurs at  $\lambda_w \cong 40 \mu$ . This wavelength corresponds to the maximum radiation intensity at 73° K, i. e., to the abscissa 0.15 in Fig. 3. Thus, the experimental data are consistent with the theory that radiation scattering by white soot reaches a maximum at low temperatures.

#### NOTATION

$a$  is the thermal diffusivity;  $c$  and  $c_c$  are the specific heats of sample and core;  $d$  is the particle diameter;  $E$  is the modulus of elasticity;  $k$  is a constant;  $L$  is

the length of cylindrical calorimeter;  $m$  is the cooling rate;  $n$  is the refractive index;  $p$  is the specific load;  $R$  and  $R_c$  are the radii of the shell and core of the calorimeter, respectively;  $T_0$  is the temperature in center of sample;  $T_1$  and  $T_2$  are the boundary temperatures:  $\Delta T = T_1 - T_2$ ;  $T_{\text{av}} = (T_1 + T_2)/2$ ;  $\gamma$  and  $\gamma_c$  are the densities of the sample and core;  $\gamma_s$  is the density of integral material;  $\epsilon$  is the porosity;  $\lambda$  is the apparent thermal conductivity of insulating material;  $\lambda_s$  is the thermal conductivity of monolithic material;  $\lambda_{\text{con}}$  is the contact thermal conductivity;  $\lambda_{\text{rad}}$  is the radiation thermal conductivity;  $\lambda_w$  is the wavelength of radiation;  $\rho$  is the phase shift.

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