

A METHOD OF DETERMINING THE THERMAL CONDUCTIVITY OF POWDERED AND FIBROUS INSULATION AS A FUNCTION OF FILLER GAS PRESSURE.

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An examination is made of the theoretical basis and implementation of a nonstationary method of rapid measurement of the thermal conductivity of powdered and fibrous insulation under conditions of monotonic change of filler gas pressure.

The thermal conductivity of powdered and fibrous materials is measured mainly by stationary methods [1-5], and to some extent by the method of monotonic heating at constant pressure [6, 7], which differs from the stationary methods as regards the duration of the experiment, since it permits determination in one experiment of the dependence $\lambda(t)$ over a wide temperature range. Experience shows, however, that the thermal conductivity of the above materials has a more complex dependence on the pressure of the filler gas than on temperature. It may therefore sometimes be of great practical interest to have a method of rapid determination of thermal conductivity as a function of pressure at constant temperature. Methods of this type should facilitate and shorten the investigation of thermal conductivity as a function of temperature and pressure. In addition, it is considerably simpler to thermostat porous materials at variable pressure than to keep the pressure steady at variable temperature.

The calorimetric equipment employed in the above method is shown in Fig. 1, and consists of a solid airtight thermostatted metal shell with a metal core mounted concentrically inside it. The gap between the two is uniform and is filled with the material to be tested. At the points C and S the sensitive junctions of thermocouples are installed, the "cold" junctions being thermostatted.

Before the start of the experiment, the electrical heater is used to achieve a prescribed temperature of the core over the shell. The test then proceeds under conditions of free cooling of the core (with the heater off) through the layer of test material completely surrounding it. The cooling stage is accompanied by a quite slow variation of the pressure of the gas filling the working cavity of the calorimeter. The pressure (or vacuum) is recorded continuously. The thermal conductivity of the material as a function of pressure is computed from the values, measured continuously during the test, of rate of cooling of the core and of its excess temperature relative to the shell. The heat flux is calculated from the known heat capacity of the core. A series of tests at various values of shell temperature are made in order to study the complex dependence $\lambda(H, t)$.

The calorimeter, which is of optimum design in order to simplify the calculations, must satisfy definite conditions.

1. The temperature field in the core and in the shell must be practically uniform.
2. The pressure during the test must be varied very slowly and be equalized within the test material.
3. The thickness of the test layer must be small enough to satisfy the inequality $c \leq 0.1 c_C$, in which case the layer may be assumed to be practically plane with effective surface area $F = (F_C + F_S)/2$.
4. The excess temperature of the core over the shell, ϑ_C , should not exceed 20-50 degrees, in the test, and therefore the thermophysical properties of the test material within the temperature drop ϑ_C , to sufficient accuracy (not worse than $\pm 1.0\%$), conform at constant pressure to a linear relation of the type

$$\lambda = \lambda_0(1 + k_\lambda \vartheta), \text{ where } |k_\lambda \vartheta| \leq 0.1,$$

$$\mu = a_0(1 + k_a \vartheta), \text{ where } |k_a \vartheta| \leq 0.1.$$

Thus, during the test the metal core cools through a thermally insulating layer of low heat capacity and with thermostatted outer surface. The temperature field within the layer may then be considered as plane, one-dimensional, and nearly steady. The formula for calculating $\lambda(H)$ of the layer may be obtained from the condition of heat transfer between core and layer at the boundary with the shell ($x = 0$):

$$\lambda_s = -c_c b_c \left(1 + \frac{c}{c_c} \frac{b_v}{b_c}\right) / F \left(\frac{\partial \vartheta}{\partial x}\right)_s \quad (1)$$

Formula (1) contains parameters $(\partial \vartheta / \partial x)_0$ and b_v , which are not measured directly in the test. They may be related with sufficient accuracy, however, to the measured parameters ϑ_C / δ and b_c through an approximate analysis of the temperature field within the test layer.

The equation of heat conduction of the layer,

$$\frac{\partial}{\partial x} \left(\lambda \frac{\partial \vartheta}{\partial x} \right) = c \gamma \frac{\partial \vartheta}{\partial \tau},$$

may be transformed, in virtue of conditions 3 and 4, to an approximation of the form

$$\frac{\partial^2 \vartheta}{\partial x^2} = -k_\lambda \left(\frac{\partial \vartheta}{\partial x}\right)^2 + \frac{1}{a_0} \frac{\partial \vartheta}{\partial \tau} \quad (2)$$

(omitting terms of higher orders of smallness).

The terms appearing on the right of the equation are corrections, since, as a whole, they determine the curvature of the temperature field in the layer which is a small quantity. They may be estimated using the results of solving (2) in the "zero" approximation

$$\frac{\partial^2 \vartheta^0}{\partial x^2} = 0,$$

whence, taking account of the boundary conditions

$$\vartheta(0, \tau) = 0, \quad \vartheta(\delta, \tau) = \vartheta_c(\tau) \quad (3)$$

we have

$$\left(\frac{\partial \vartheta^0}{\partial x}\right)^2 = \left(\frac{\vartheta_c}{\delta}\right)^2, \quad \frac{\partial \vartheta^0}{\partial \tau} = b_c \frac{x}{\delta}. \quad (4)$$

The solution of (2) in the "first" approximation, taking (3) and (4) into account, gives a more accurate expression for the temperature field in the layer

$$\vartheta = \frac{\vartheta_c}{\delta} x \left(1 + \frac{1}{2} k_\lambda \vartheta_c - \frac{1}{6} \frac{b_c \delta^2}{a_0 \vartheta_c}\right) - \frac{1}{2} k_\lambda \left(\frac{\vartheta_c}{\delta}\right)^2 x^2 + \frac{1}{6} \frac{b_c x^3}{a_0 \delta},$$

whence the desired relations for $(\partial \vartheta / \partial x)_0$ and b_V may be written in the form

$$\left(\frac{\partial \vartheta}{\partial x}\right)_0 = \frac{\vartheta_c}{\delta} \left(1 + \frac{1}{2} k_\lambda \vartheta_c - \frac{1}{6} \frac{b_c \delta^2}{a_0 \vartheta_c}\right), \quad b_V \approx \frac{1}{2} b_c \quad (5)$$

Allowance has been made in (5) for the fact that $b_V(\tau)$ appears as a correction term in (1), and affords a better estimate than does $(\partial \vartheta / \partial x)_0$.

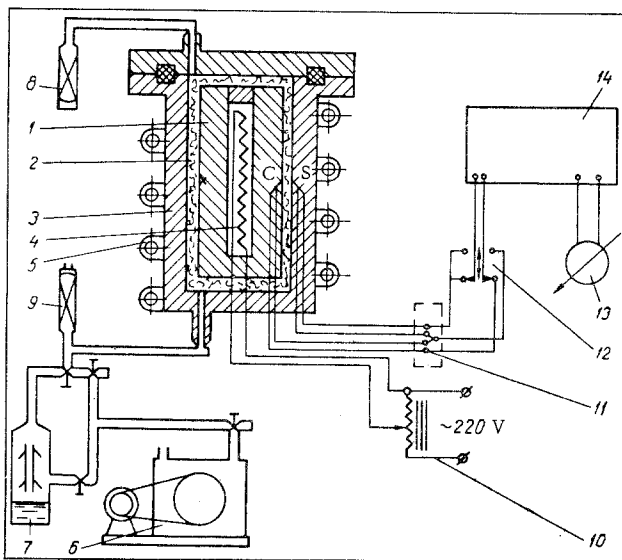


Fig. 1. Principal features of the equipment: 1) core; 2) test material; 3) shell; 4) heater; 5) coil; 6) fore-vacuum pump; 7) diffusion pump; 8), 9) LT-2 thermocouple bulbs; 10) LATR-1 autotransformer; 11) "cold" junction unit; 12) switch; 13) galvanometer; 14) potentiometer.

After substitution of (5) into (1) and certain simplifying transformations of the structure of the correction terms

$$\frac{b_c \delta^2}{a_0 \vartheta_c} \approx -\frac{c}{c_c}, \quad \lambda_0 \left(1 + \frac{1}{2} k_\lambda \vartheta_c\right) = \bar{\lambda}$$

the final form of the formula for calculating $\bar{\lambda}$ is

$$\bar{\lambda} = \frac{|b_c|}{\vartheta_c} \frac{c_c \delta}{F} \left(1 + \frac{1}{3} \frac{c}{c_c}\right).$$

To verify the method experimentally, and to further investigate the thermal conductivity of powders and fibrous materials in low-pressure conditions, the equipment of Fig. 1 was set up.

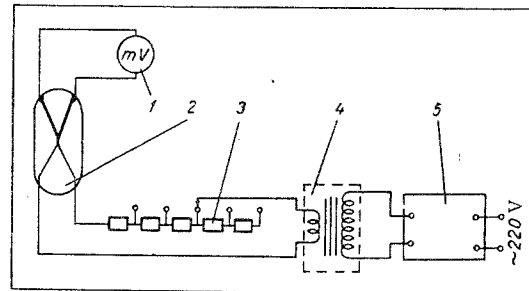


Fig. 2. Electrical circuit of vacuum-meter: 1) M-82 millivoltmeter; 2) LT-2 thermo-couple bulb; 3) R-14 resistance box; 4) 220/5.5-V transformer; 5) ST-250 stabilizer.

Its main elements are: a) calorimeter unit; b) a vacuum system with RVN-20 fore-vacuum pump and TsVL-100 diffusion pump; c) an electrical supply for the core heater with autotransformer; d) a vacuum measuring system with two thermoelectric sensors; e) a temperature measuring system, including two nichrome/constantan thermocouples S and C of diameter 0.2 mm, a type PPTN-1 laboratory potentiometer with a galvanometer and a "cold" junction unit.

The core and the shell of the calorimeter were made of brass. The thickness of the test layer was 6 mm, the core diameter 50 mm, height 114 mm, and mass 1.97 kg. The heater was spiral in form, mounted in a porcelain tube; its heat capacity was about 0.1% of that of the core and was not taken into account in the calculations.

The vacuum system could reach a limiting level of the order of 10^{-1} N/m², which was quite satisfactory for the study of powdered and fibrous materials, since at $H < 10^{-1}$ N/m² the conductivity λ of such materials is independent of pressure [2-5]. The rate of evacuation of air from the calorimeter was controlled manually using two valves in series.

The strong dependence of thermal conductivity of porous materials on pressure (or vacuum) in the range 10^4 - 10^{-1} N/m² necessitated continuous and quite accurate measurement of pressure within the test material during the entire test. The total range of pressure measurement in these tests should be

$10^5 - 10^{-2} \text{ N/m}^2$. Industrial vacuum-meters proved unsuitable for this purpose, since they usually operate in a narrow pressure range, and also either do not attain the required accuracy or are excessively slow and do not permit continuous measurement.

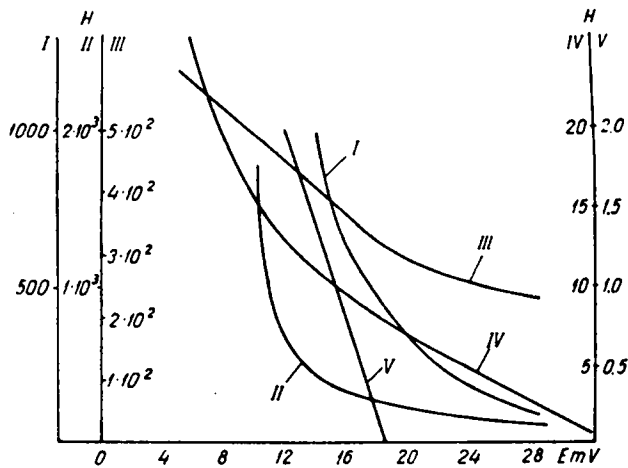


Fig. 3. Calibration curves of the LT-2 thermocouple bulb (I, II, III, IV, V—pressure ranges of filler gas).

For the pressure measurements, therefore, we constructed a forced-heating thermocouple vacuum-meter (Fig. 2), the operating principle of which is described in [8]. In [8] the filament of the LT-2 bulb was maintained at a constant temperature of the order of 800°C , the measured parameter being filament supply current. In our vacuum-meter circuit a different operating regime of the LT-2 bulb proved to be expedient. A stabilized voltage was supplied to the filament, and the pressure was measured in terms of filament temperature, i. e., from the readings of a thermocouple attached to it. This regime was simpler to accomplish, stabilization being achievable with any industrial voltage stabilizer. An EPP-09 potentiometric recorder may be used for automatic and continuous recording of the vacuum-meter readings.

The total range of pressure measured by the bulb was divided into five ranges: $10^5 - 2 \cdot 10^3 \text{ N/m}^2$; $2 \cdot 10^3 - 2 \cdot 10^2 \text{ N/m}^2$; $6 \cdot 10^2 - 2 \cdot 10 \text{ N/m}^2$; $30 - 2 \text{ N/m}^2$ and $2 - 5 \cdot 10^{-2} \text{ N/m}^2$. In going from one range to another, the bulb supply voltage was changed in steps. The supply voltage values and the boundaries between ranges were chosen experimentally in such a way that the thermocouple bulb emf varied in the limits 10–32 mV, corresponding roughly to filament temperature variation from 250° to 500°C . The bulb should be calibrated with a reference vacuum-meter. In particular, in the pressure range $10^5 - 2 \cdot 10^3 \text{ N/m}^2$, the bulb may be calibrated with a U-tube mercury manometer, and in the range $2 \cdot 10^3 - 5 \cdot 10^{-2} \text{ N/m}^2$ with a standard mercury-glass McLeod manometer. The accuracy of calibration in the various ranges varied from ± 20 to $\pm 50\%$ as the pressure dropped,

the error being determined mainly by the error of the reference manometer. Calibration curves of one of the LT-2 bulbs are shown in Fig. 3. Two bulbs were used in the experimental equipment (Fig. 1): one ahead of the calorimeter, the other behind it, which afforded a measure of the uniformity of pressure distribution within the test material.

The equipment was used to measure the thermal conductivity of quartz sand and lead shot, and the experimental curves are shown in Fig. 4. The tests were run at room temperature, and the initial excess temperature of the core was about 30 degrees. The error in the measured values of $\lambda(H)$ was about $\pm 5\%$, and the over-all error, including pressure estimation, about $\pm 10\%$.

The tests showed that the thermal conductivity of finely disperse powdered materials in the high-vacuum region depends very substantially on the time spent by the material at the given pressure, this being evidently due to the presence on the particles of material of a hard-to-evaporate film of absorbed air, or sometimes oil. For this reason it is expedient to carry out the tests under conditions of smooth increase of pressure in the calorimeter with prolonged preliminary soaking at the limiting vacuum, it being also expedient to "anneal" the material.

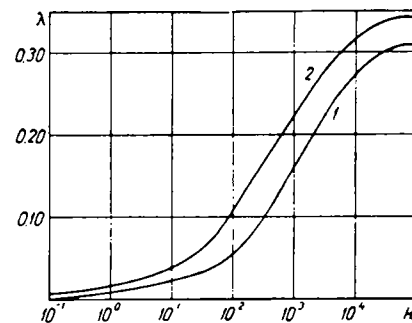


Fig. 4. Dependence of effective thermal conductivity of test material on pressure of filler gas: 1) quartz sand, $d = 0.2 \text{ mm}$, $p = 35\%$, $\gamma = 1700 \text{ kg/m}^3$; 2) lead shot, $d = 1.25 \text{ mm}$, $p = 35\%$, $\gamma = 7400 \text{ kg/m}^3$.

NOTATION

t —temperature; λ, α —thermal conductivity and diffusivity of test material; k_λ, k_α —relative temperature coefficients of λ and α ; δ —thickness of test layer; x —variable layer coordinate reckoned from shell; $\delta = \delta(x)$, δ_c —excess temperature of material at section x and of core over shell; b_c, b_v —rate of cooling of core and of variation of volume-mean temperature of layer; c_c, c —total heat capacity of core and material; F_s, F_c —area of working surfaces of shell and core; d —diameter of particles of bulk material; p —material porosity; γ —volume density of material.

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