at 1200°K, and above this temperature its resistance is smaller than that of the purer metal. This behavior of ρ of the cobalt sample investigated apparently results from a decrease in the magnetic component of the resistance due to impurities [12].

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COMPARATIVE METHOD FOR MEASURING THERMAL CONDUCTIVITY

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UDC 536.2.08

A comparative method is developed for measuring the thermal conductivity of solid and dispersed materials with λ = 0.1-80 W/(m · °K). The method is accurate, rapid, and simple to use.

As the number of standard materials increases, comparative methods of measuring thermophysical characteristics are more and more widely used. As a rule, these methods involve the measurement of a smaller number of parameters, eliminate certain systematic errors, and are as accurate as absolute methods.

The method proposed is simple to use, is highly accurate, and can be used to determine the thermal conductivity of various materials with $\lambda = 0.1 - 80$ W/(m · °K). Since the experiment is of short duration, the method can be used to measure the thermal conductivity of moist materials.

A schematic diagram of the method is shown in Fig. 1a. The sample 1 in the form of a plate and contiguous heat meter 2 are placed between two massive metal blocks 3 with the same heat capacity. The lateral surface of the blocks, the sample, and the heat meter are surrounded by ideal thermal insulation 4. The temperature of one of the blocks, for example, the upper one, is raised 5-10°K above that of the lower. After a certain time a nearly steady heat flux is established between the blocks; this flux depends on the initial temperature difference between the upper and lower blocks and the thermal resistance of the sample under study. If the heat capacities of the blocks are large enough, their temperatures remain practically

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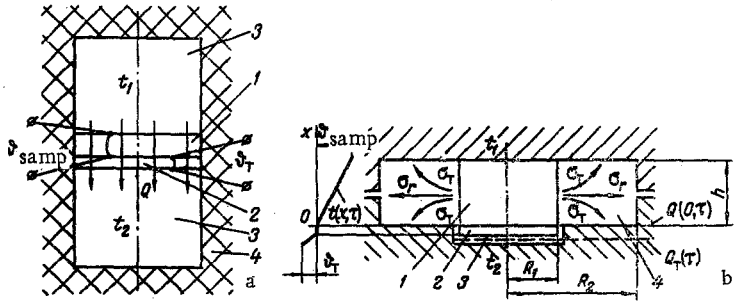

Fig. 1. Schematic diagram of method: a) thermal model of method; b) heat exchange in an annular sublayer.

constant during the experiment. The experiment consists in measuring the temperature drop ϑ_{samp} across the sample and ϑ_T across the heat meter. The thermal conductivity λ is calculated from the expression

$$\lambda = \frac{k_T h}{S} \cdot \frac{\vartheta_T}{\vartheta_{samp}} = \frac{k_T h}{S} \cdot \frac{N_T}{N_{samp}}. \tag{1}$$

Under actual conditions, however, the temperatures of the blocks change because of heat flow between them. To obtain quantitative limitations on the experimental conditions we consider the temperature distribution in an infinite plate $0 < x < h$ for a linear variation of the temperature of its surfaces.

The heat-conduction equation has the form

$$\frac{\partial t}{\partial \tau} = a \frac{\partial^2 t}{\partial x^2} \tag{2}$$

and the boundary conditions are

$$\begin{aligned} \varphi_1(\tau) &= t(0, \tau) = t_1 + b\tau; \\ \varphi_2(\tau) &= t(h, \tau) = t_2 - b\tau; \\ F(x) &= t(x, 0) = t_1. \end{aligned} \tag{3}$$

The problem was solved by Taits [1] for arbitrary functions $\varphi_1(\tau)$, $\varphi_2(\tau)$, and $F(x)$:

$$t(x, \tau) = \varphi_1(\tau) + \frac{\varphi_2(\tau) - \varphi_1(\tau)}{h} + \frac{2}{h} \sum_{l=1}^{\infty} \sin\left(\varepsilon \frac{x}{h}\right) \exp\left(-\varepsilon^2 \frac{a\tau}{h^2}\right) \times$$

$$\times \int_{-h}^{h} \left[F(x) - \varphi_1(0) - \frac{\varphi_2(0) - \varphi_1(0)}{h} x\right] \sin\left(\varepsilon \frac{x}{h}\right) dx +$$

$$+ \sum_{l=1}^{\infty} \frac{2}{\varepsilon} \sin\left(\varepsilon \frac{x}{h}\right) \exp\left(-\varepsilon^2 \frac{a\tau}{h^2}\right) \int_0^{\tau} \exp\left(\varepsilon^2 \frac{a\tau}{h^2}\right) [\varphi_2'(\tau)(-1)^l - \varphi_1'(\tau)]\, d\tau. \tag{4}$$

We obtain the required solution from (4) by using the boundary conditions (3):

$$t(x, \tau) = t_1 + b\tau + \frac{t_2 - t_1}{h} x - \frac{2b\tau}{h} x + \frac{2bh^2}{a} \Phi_1 - 4(t_2 - t_1)\Phi_2,$$

where

$$\Phi_1 = \sum_{l=1}^{\infty} \frac{[1+(-1)^l]}{\varepsilon^3} \sin\left(\varepsilon \frac{x}{h}\right)\left[\exp\left(-\varepsilon^2 \frac{a\tau}{h^2}\right) - 1\right],$$

$$\Phi_2 = \sum_{l=1}^{\infty} \frac{1}{\varepsilon^2} \sin\left(\varepsilon \frac{x}{h}\right) \exp\left(-\varepsilon^2 \frac{a\tau}{h^2}\right). \tag{5}$$

For sufficiently large Fo = $a\tau/h^2$ the terms containing exp $[-\varepsilon^2(a\tau/h^2)]$ will tend to zero. For certain values of Fo > Fo_{st} a quasistationary stage of the process begins, described by the equation

$$t(x, \tau) = t_1 + b\tau + \frac{t_2 - t_1}{h} x - \frac{2b\tau}{h} x + \frac{bx^2}{2a} - \frac{bx^3}{3ah} - \frac{bhx}{6a}. \tag{6}$$

We find the distribution of heat fluxes of the plate from Eq. (6),

$$q(x, \tau) = -\lambda \frac{dt}{dx} = -\lambda\left[\frac{t_2 - t_1}{h} - \frac{2b\tau}{h} + \frac{bx}{a} - \frac{bx^2}{ah} - \frac{bh}{6a}\right]. \tag{7}$$

The heat flux passing through the sample is measured by the metal heat meter (Fig. 1b).

Taking account of the fact that the heat meter measures the flux in the middle of the working layer, the flux Q(0, τ) emerging from the sample is related to the flux $Q_T(\tau)$ by the expression

$$Q(0, \tau) = Q_\tau(\tau) + Q_{abs}(\tau), \tag{8}$$

where

$$Q(0, \tau) = q(0, \tau) S,\ Q_\tau(\tau) = k_\tau \vartheta_\tau(\tau),\ Q_{abs}(\tau) = C_p b_p + 0.5\, C_\tau b_\tau \approx (C_p + 0.5\, C_\tau) b.$$

The last equation is valid, since there is good thermal contact between the sample and the plate, and the temperature distribution of the heat meter plate is uniform and $C_p \gg 0.5\ C_T$.

According to (7) the heat flux across the lower boundary of the sample is

$$Q(0, \tau) = q(0, \tau) S = \frac{\lambda S}{h}\left(t_2 - t_1 - 2b\tau - \frac{bh^2}{6a}\right).$$

Taking account of the fact that

$$t(0, \tau) = t_1 + b\tau,\ t(h, \tau) = t_2 - b\tau,$$
$$\vartheta_{samp}(\tau) = t(h, \tau) - t(0, \tau),$$

we obtain

$$Q(0, \tau) = \frac{\lambda S}{h}\left[\vartheta_{samp}(\tau) - \frac{bh^2}{6a}\right].$$

From Eq. (8) we have

$$\frac{\lambda S}{h} \vartheta_{samp}(\tau) - \frac{\lambda S b h^2}{6ah} = (C_p + 0.5\, C_\tau) b + k_\tau \vartheta_\tau(\tau),$$

from which by using the relation $\lambda = a c\rho$ we obtain the working equation

$$\lambda = \frac{[k_T \vartheta_T(\tau) + C_\Sigma b] h}{S \vartheta_{samp}(\tau)}, \quad C_\Sigma = C_p + 0.5 C_T + 0{,}16 C. \tag{9}$$

If heat transfer between the blocks is mainly through the sample, and heat transfer between the blocks and the medium is relatively small, the relation

$$k_T \vartheta_T \approx C_{BL} b,$$

holds, and using this Eq. (9) takes the form

$$\lambda = \frac{k_T h}{S} \cdot \frac{\vartheta_T(\tau)}{\vartheta_{samp}(\tau)} \left(1 + \frac{C_\Sigma}{C_{BL}}\right). \tag{10}$$

If in Eqs. (9) and (10)

$$k_T \vartheta_T \gg C_\Sigma b \quad \text{or} \quad \frac{C_\Sigma}{C_{BL}} \ll 1, \tag{11}$$

then the working formula (1) follows from the initial equations.

Equation (11) must be taken into account in designing the calorimeter.

In carrying out the method it is very important to determine the duration of the initial idle stage of the experiment. Using the fact that condition (11) can practically always be satisfied, an estimate was made on the basis of a solution of Eq. (2) for the boundary conditions

$$t(0, \tau) = t_1, \; t(h, \tau) = t_2, \; t(x, 0) = t_1.$$

The solution of Eq. (2) for the boundary conditions indicated has the form

$$t(x) = t_1 + (t_2 - t_1) \frac{x}{h} - (t_1 - t_2) \sum_{l=1}^{\infty} \frac{2(-1)^l}{\varepsilon} \sin\left(\varepsilon \frac{x}{h}\right) \exp(-\varepsilon^2 \mathrm{Fo}).$$

Since the heat flux was measured at the lower edge of the sample at x = 0, the duration of the transient stage is determined by the value of Fo_{st} for which with an error δ_{adm} the terms containing the factor $\exp(-\varepsilon^2 \mathrm{Fo})$ in the expression for the flux $q(0, \tau)$ can be neglected,

$$\frac{dt}{dx} = \frac{t_2 - t_1}{h} + (t_2 - t_1) \sum_{l=1}^{\infty} \frac{2(-1)^l}{\varepsilon} \cos\left(\varepsilon \frac{x}{h}\right) \frac{\varepsilon}{h} \exp(-\varepsilon^2 \mathrm{Fo});$$

$$\left.\frac{dt}{dx}\right|_{x=0} = \frac{t_2 - t_1}{h} \left[1 + \sum_{l=1}^{\infty} 2(-1)^l \exp(-\varepsilon^2 \mathrm{Fo})\right].$$

Limiting ourselves to the first term of the series we have

$$-2 \exp(-\pi^2 \mathrm{Fo}_{st}) \leqslant \delta_{adm},$$

from which

$$\mathrm{Fo}_{st} \geqslant \ln \frac{\delta_{adm}}{2} \Big/ \pi^2 \quad \text{or} \quad \mathrm{Fo}_{st} \geqslant 0.1 \left|\ln \frac{\delta_{adm}}{2}\right|. \tag{12}$$

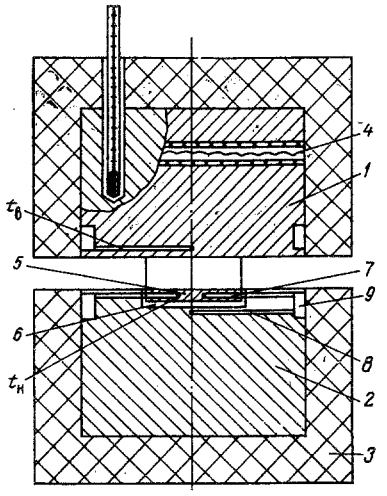

Fig. 2. Design of mockup of calorimeter.

Equation (12) should be used in the design of the calorimeter.

Calculations and experiment show that the time to reach the steady state for δ_{adm} = 1% lies in the range τ_{st} = 1 = 8 min for samples 10 mm thick. In performing the experiment this time is easily determined from the time to establish a signal N_T from the heat meter.

The lateral surface of the sample is insulated by an annular sublayer (Fig. 1b). The layer can be filled with air or an efficient heat insulator such as porolon or foam plastic.

The error due to lateral heat transfer of the sample can be determined by the relation

$$\delta Q_{leak} = \frac{Q_{leak}}{Q_T} = \frac{Q_{leak}}{k_T \vartheta_T}.$$

The flux $\delta(Q_{leak})$ leaking from the lateral surface of the sample is determined by the algebraic sum of the fluxes to the lower and upper end and the outer annular surfaces of the sublayer,

$$Q_{leak} = \sigma_T (t_2 - \bar{t}_{samp}) + \sigma_T (t_1 - \bar{t}_{samp}) + \sigma_r (\bar{t}_{BL} - \bar{t}_{samp}). \quad (13)$$

The thermal conductivities σ_T and σ_r are given in [2].

Taking account of the fact that

$$t_2 - t_1 = \vartheta_{samp} + \vartheta_T, \quad \bar{t}_{samp} = t_2 - \frac{\vartheta_{samp}}{2}, \quad \bar{t}_{BL} = \frac{t_1 + t_2}{2},$$

we obtain the value of the required error,

$$\delta Q_{leak} = -\frac{(\sigma_T + 0.5\,\sigma_r)\vartheta_T}{k_T \vartheta_T} = -\frac{\sigma_T + 0.5\,\sigma_r}{k_T}.$$

Analysis shows [2] that for $h/R_1 \leqslant 0.5$ and $R_2/R_1 \geqslant 3$ the conductivity $\sigma_r = 0$ and the leakage flux are determined solely by the asymmetry of the heat transfer between the lateral surface of the sample and the adjoining end surfaces of the annular sublayer:

$$\delta Q_{leak} = -\frac{\sigma_T}{k_T}. \quad (14)$$

This is an interesting result, since the error under consideration does not depend on the thermal resistance of the sample under study for a constant thickness h but is determined solely by the geometrical parameters of the calorimeter itself.

To chose the material and mass of the blocks it is necessary to know the time to establish a steady state in the blocks themselves. To this end the solution of the heat-conduction problem for an infinite plate was analyzed for a constant heat flux density incident on its surface and convection boundary conditions at the opposite face [3]. Calculation shows that by using metal blocks it is easy to obtain values of Bi < 0.1, and for such values a steady state is established for Fo > 0.25.

Beginning with Fo > 0.25 the whole heat capacity of the blocks takes part in the absorption of the heat flux from the sample and its value can be chosen on the basis of Eq. (11).

The method was checked on the mockup shown in Fig. 2. The mockup consists of two identical Duralumin blocks 1 and 2 thermally insulated by a shell of foam plastic 3. There is an electric heater 4 in the upper block. The contact surfaces of the blocks are ground and have a ▽8 surface finish. A metal heat meter consisting of a contact copper plate 5, 2 mm thick and 0.3-mm-thick epoxy resin working layer of the heat meter 6 is mounted at the contact surface of the lower block. The lateral gap around the heat-meter plate was also filled with epoxy resin which is included in the thermal conductivity of the heat meter k_T. A total of 12 radial holes 7 were drilled in the heat-meter plate in which were mounted a thermocouple junction t_H and 11 heat-meter thermopile junctions. The other 11 junctions of the heat meter were mounted in radial holes 8 of the lower block close to the working layer of the heat meter. The thermocouples were led out of the heat-meter plate through radial slots to the annular channel 9. The junctions of the heat-meter thermopile were connected in the annular channel and then the radial and annular channels were filled with epoxy resin. A single thermocouple t_B was mounted at the contact surface of the upper block. The thermocouples t_H and t_B are connected to a differential thermocouple which measures the temperature drop ϑ_{samp} across the sample. All the thermocouples are connected to the block of cold junctions, and connections to the outside are made through a stranded copper wire. The measurements were made with a type M195/1 mirror galvanometer. The temperature of the upper block is recorded by a mercury thermometer.

Measurements of dispersed materials are made in a sealed cell with base and lid of industrial copper and side walls of 0.5-mm-thick polymethyl methacrylate. The lid and base have 1-mm-diameter holes for the junctions of an armored differential thermocouple. It is recommended that this same thermocouple be used to measure the temperature drop across samples with $\lambda > 5 W/(m \cdot °K)$.

The heat meter was calibrated after the calorimeter was assembled. Fused quartz and polymethyl methacrylate were used as standard samples [4]. The thermal conductivity of the heat meter k_T was calculated from Eq. (1).

The use of 2-10-mm-thick samples for calibration eliminated the effect of contact resistances. The values of k_T obtained with quartz and polymethyl methacrylate agreed within 1%, indicating no systematic errors related to changes in the thickness and the thermal conductivity of the standard samples.

Calculations performed for the mockup show that the error resulting from the heat capacity C_Σ is $\delta(C_\Sigma) = 1\%$, that from the leakage flux $\delta(Q_{leak}) = 0.6\%$, and contact resistance $\delta(R_c) = 0.7\%$. Taking account of the fact that the uncertainty in the values of the conductivity of the standard samples is $\delta(\lambda_s) = 2\%$, and also taking into account the instrumental errors of Eq. (1), the error in measuring the thermal conductivity of solids is no more than 3% with a confidence coefficient of 0.95.

If samples of the same thickness are used in the calibration of the heat meter and in the working experiments, the errors $\delta(C_\Sigma)$ and $\delta(Q_{leak})$ become negligible and for the measurement of the temperature drop ϑ_{samp} by the differential thermocouple mounted in the sample or lid of the cell the error $\delta(R_c)$ also becomes negligible. In this case the error is estimated as $\delta(\lambda) = 2.5\%$ with a confidence coefficient of 0.95.

A large number of solids and moist dispersed materials were investigated with the mockup.

NOTATION

τ, time; λ, thermal conductivity; b, b_p, b_T, rates of change of temperatures of block, plate, and working layer of heat meter; C_{BL}, C_p, C_T, total heat capacities of block, plate, and working layer of heat meter; S, h, cross-sectional area and thickness of sample; k_T, thermal conductivity of heat meter; N_T, N_{samp}, instrument readings in measurement of heat-meter signal and temperature drop across sample; σ_T, σ_r. thermal conductivities from lateral surfaces of sample to end and radial surfaces of annular sublayer; R_1, R_2, radii of sample and block; $\bar{t}_{samp}$, $\bar{t}_{BL}$, average temperatures of sample and blocks; $\varepsilon = \pi l$ (l = 1, 2, 3, ...); δ_{adm}, admissible error of measurement.

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DETERMINATION OF LIQUID SOLIDIFICATION PRESSURE BY MEASUREMENT OF DIELECTRIC LOSSES

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A method is described for determining solidification pressure p_s of a liquid by measuring its low-frequency dielectric losses; experimental p_s values are presented.

The problem of determining liquid solidification pressures arises from the need to protect components of high-pressure apparatuses from injury due to solidification of the working liquid or liquid being studied. This is especially important in the operation of fragile components of precision apparatuses such as high-pressure viscosimeter measurement cells, bellows piezometers, resistance manometer coils, etc., and in establishing the operating characteristics of new working fluids. This question is also of great practical importance in several branches of industry (lubricant production, hydroextrusion, etc.).

In the great majority of cases the transformation of a liquid into a solid state occurs more or less gradually and is characterized by the formation of an amorphous glass. The abrupt crystallization of metals at a fixed pressure and temperature is probably only an extremal case of the more universal phenomenon of vitrification. In vitrification the liquid viscosity gradually becomes so high that formation of a crystalline lattice is impossible due to the low mobility of molecules or atoms. Vitrification is often observed at high pressures, since the viscosity of many liquids increases by several orders under such conditions. Thus, liquid solidification at high pressure extends over a certain pressure interval for any given fixed temperature.

At present there exists no generally accepted criterion defining the completion of the transformation of the liquid phase to solid. It is probably most expedient to relate the degree of solidification of a substance to its viscosity. For example, Turnball [1] proposes that a substance be considered solidified when its viscosity exceeds 10^{14} Pa · sec. But with this criterion, tin at room temperature is not

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