DETERMINATION OF THE THERMAL CONDUCTIVITY OF PARAFFIN AT LOW TEMPERATURES

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The authors describe a method of accurately determining the thermal conductivity of insulators with small volume coefficients of thermal expansion. Results are given for an experimental determination of the thermal conductivity of paraffin in the temperature range 83-295°K.

A one-dimensional form of the thermal potentiometer method [1] has been used to determine thermal conductivity. A constant temperature gradient was set up along the axis of a cylindrical specimen with a heater and cooler attached to its ends. The substance examined was white paraffin (type A). Certain properties of paraffin affected details of the experimental set-up: a) its volume coefficient of thermal expansion, $\beta \approx 1 \cdot 10^{-4}$/degree, is large in comparison with that of metals; b) it is sensitive to thermal shock; and c) it has a high surface emissivity, $\varepsilon \approx 0.95$.

Because of the large difference in the expansion coefficients of metals and paraffins, we took special steps to achieve reliable contact between the specimen and the heater and cooler over a wide temperature range. The cross section of the specimen, and hence the area of contact, was made as small as possible, and the surfaces of the heater and cooler had projections which were fused into the end faces of the cylindrical specimen. In order to be able to neglect the resulting geometrical irregularities in calculating the conductivity, it was necessary to measure the temperature gradient along the specimen at a considerable distance from heater and cooler. The corresponding unavoidable increase in specimen length increased the duration of the experiment.

The low thermal conductivity of paraffin, its high surface emissivity, and the long length and small diameter of the specimen lead to considerable lateral heat loss by radiation. A protective ring of the same material is generally used to compensate for heat losses [2]. This is inconvenient in our case, since if the cross section of the paraffin specimen is increased, it is difficult to achieve contact between it and the heater and cooler. Increased mass is also undesirable, because large specimens are particularly subject to thermal shock which may occur in mounting and cooling the specimen.

A sharp decrease in the radiative heat loss was achieved by enclosing the specimen in a cylindrical metal screen, leaving a small gap. Temperatures of screen and specimen at the same height were kept the same by means of a supplementary heater. The polished screen reflected almost all the radiant energy flux, and itself emitted practically no radiation. Some heat transfer by radiation is, however, unavoidable in these conditions: radiation from any point on the lateral surface of the specimen, at an angle to the normal, will be reflected from the screen to strike another point on the surface. From simple physical considerations it is clear that heat transfer due to radiation will decrease with the gap between specimen and screen.

To resolve the question of the accuracy of the method, and to choose correct dimensions for specimen and screen, we examined the effect of the above heat transfer on the temperature distribution in the specimen, making certain simplifying assumptions.

Consider a cylindrical bar of radius R and length l (Fig. 1), enclosed within a concentric cylindrical screen to give a gap h. The temperature T of one end of the bar ($x = l$) is constant and equal to T_0. At the other end of the specimen a heat flux q, uniform across the section, is established. For convenience of calculation, put $\theta = T - T_0$. Under steady conditions, $\theta(x, r)$ obeys the equation

$$\frac{\partial^2 \theta}{\partial x^2} + \frac{1}{r} \frac{\partial}{\partial r} \left(r \frac{\partial \theta}{\partial r} \right) = 0. \tag{1}$$

The boundary conditions at the ends of the specimen have the form

$$\theta \Big|_{x=l} = 0; \quad \lambda \frac{\partial \theta}{\partial x} \Big|_{x=0} = q. \tag{2}$$

To simplify derivation of the boundary condition at the sides, assume that the specimen is a perfect black body, and that the inside walls of the screen (sections AB, BC, CD) wholly reflect the radiant energy incident upon them (reflection at the screen walls assumed to be specular). The assumption of this actually non-existant reflection from the ends AB and CD appreciably simplifies the solution, and for $h \ll l$ clearly cannot affect the end result.

Let us also assume that the temperature drop along the specimen is small compared to the mean temperature, and, finally, that the distance between the screen and specimen is considerably less than the specimen radius.

The last assumption allows an approximation for the energy radiated from unit specimen surface in unit time in the form

$$J(\vartheta, x)\, d\vartheta = \frac{1}{2} \sigma T^4(x) \cos\vartheta\, d\vartheta.$$

Since the temperature drop is small, this condition can be linearized to give

$$J(\vartheta,\ x)\, d\vartheta = \frac{1}{2}\,[A + B\theta(x)]\,\cos\vartheta\, d\vartheta,$$

where $A = \sigma T_0^4$; $B = 4\sigma T_0^3$.

To establish the desired condition at the lateral surface of the specimen, we now equate the energy reaching the surface from within the specimen to the difference between emitted and absorbed radiant energy.

$$-\lambda \frac{\partial\theta(x)}{\partial r}\bigg|_{r=R} = B\theta(x)\bigg|_{r=R} - \frac{B}{4h}\int\limits_{-\infty}^{\infty} \theta(x')\bigg|_{r=R} \frac{dx'}{\left[1 + \left(\frac{x-x'}{2h}\right)^2\right]^{3/2}}.$$

The infinite limits in this integral stem from the assumption that the walls of the evacuated housing are specular. It can be shown that the radiant energy reflected repeatedly from walls AB, BC, and CD, and absorbed in section AD, can be considered as coming from various parts of an infinitely long specimen, whose temperature is related in a simple way to the temperature T(x, r) in the section $0 < x < l$. In the section $-l < x < 0$, T(x, r) is a smooth extension of specimen temperature, $\theta(-x, r) = \theta(x, r)$, and throughout the length of the infinite rod it is a periodic function of x, with period $2l$, $\theta(x+2l, r) = \theta(x, r)$.

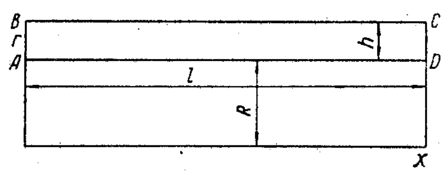

Fig. 1. Diagram of problem.

Writing the correction to the temperature distribution $\tau(x, r)$ due to radiant heat conduction along the vacuum housing as

$$\theta(x, r) = \left(1 - \frac{x}{l}\right)\frac{ql}{\lambda} + \tau(x, r) \quad \text{for} \quad 0 \leqslant x \leqslant l$$

or

$$\theta(x, r) = \frac{8ql}{\pi^2\lambda} \sum_{n=0}^{\infty} \frac{\cos[(2n+1)\pi x/2l]}{(2n+1)^2} + \tau(x, r) \text{ for any x,}$$

and separating the variables in the equation for $\tau(x, r)$, we have

$$T = T_0 + \frac{ql}{\lambda}\left(1 - \frac{x}{l}\right) - \frac{8Bql}{\lambda\pi^2} \sum_{k=0}^{\infty} \frac{1}{(2k+1)^2} \times$$
$$\times \frac{[1 - 2\eta_k hK_1(2\eta_k h)]\, I_0(\eta_k r)\cos\eta_k x}{\lambda\eta_k I_1(\eta_k R) + BI_0(\eta_k R)[1 - 2\eta_k hK_1(2\eta_k h)]}, \qquad (3)$$

where $\eta_k = \frac{2k+1}{2l}\pi$.

The second term in (3) gives the temperature distribution along the specimen in the absence of heat transfer due to radiation, and the series takes into account changes in the temperature distribution arising from thermal flux between specimen walls and screen. Expression (3) is unwieldy and unsuitable for calculating the coefficient of thermal conductivity. We can, however, use it to establish experimental conditions in which the contribution of radiation will be negligible, while we calculate the thermal conductivity from

$$\lambda_p = q \Big/ \frac{dT}{dx}. \tag{4}$$

From the need for reliable thermal contact between the specimen and the heater and cooler to preserve one-dimensional conditions, and also owing to considerations of assembly and the desire to minimize errors, we made the following choice of geometrical details: specimen diameter 2R = 2 cm, length l = 5 cm, and gap size h = 0.07 cm. It may be seen from Fig. 2 that, at the highest test temperature (295°K), the error does not exceed 2%. As the temperature falls, $(\lambda - \lambda_p)/\lambda$ decreases practically in proportion to T_0^3.

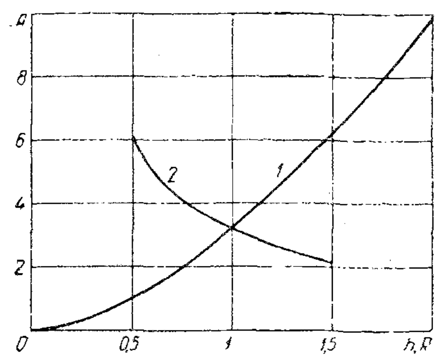

Fig. 2. Deviation of true value of λ from that calculated from formula (4), A = $= (\lambda - \lambda_p)/\lambda$ (%); as a function of:
1 – gap size h(mm) between specimen and screen, for specimen radius R = 1 cm and temperature T_0 = 300°K;
2 – specimen radius R(cm), for h = 1 mm and T_0 = 300°K.

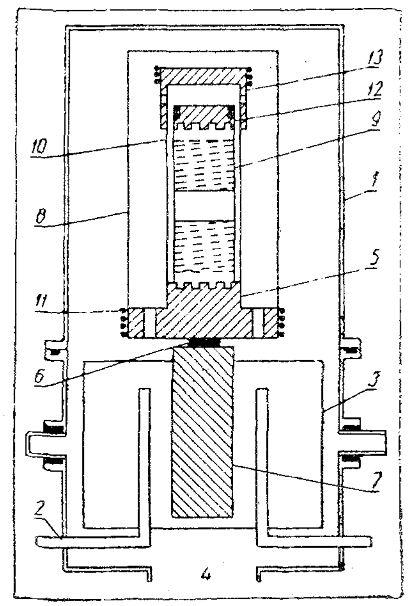

Fig. 3. Schematic of experimental apparatus: 1 – vacuum chamber; 2 – thin-walled German silver tube; 3 – flask for liquid nitrogen; 4 – vacuum pump inlet; 5 – cooler; 6 – low-conductivity spacer; 7 – massive copper bar; 8 – copper screen; 9 – specimen; 10 – inner screen; 11 – cooler coil; 12 – specimen heater; 13 – screen heater.

Fig. 3 is a schematic of the apparatus used to measure the thermal conductivity of paraffin.

By suitable choice of the thickness and material of the spacer and of the current in the heater coil, the temperature of the cooler and the outer screen was varied in the range 80-293°K, and kept constant correct to ± 0.01°K. The distance between the junctions of the differential thermocouples, used to measure the temperature gradient along the specimen axis, was 2 cm. The magnitude of the gradient did not exceed 1°K/cm. Calculation according to (3) shows that a possible shift of the thermocouple junctions in a plane perpendicular to the specimen axis has a negligibly small influence on the experimental results. To reduce heat flow along the lead-in wires, the thin (0.06 mm) copper wires of the differential thermocouple were wound on the specimen in a spiral and fused into the paraffin. For control we used two auxiliary thermocouples measuring the difference in temperature between the specimen heater and cooler, and between the heater of the specimen and that of the screen. The inside surface of the screen heater was polished, and its temperature coincided within 0.01°K with that of the specimen heater. All measurements of temperature were made with copper-constantan thermocouples. The chemical uniformity of the wires for these was carefully checked. To exclude parasitic thermal emf's the thermocouple wires were connected directly to the measuring potentiometer circuit. Null readings of the differential thermocouple with the specimen and screen heaters switched off proved the absence of parasitic thermal emf's at various cooler temperatures. The leads going to the specimen were connected in a thermal sense to the screen. The whole apparatus was shielded from environmental radiation by being enclosed in an auxiliary copper screen. The apparatus was evacuated a pressure of the order of $13.322 \cdot 10^{-7}$ newton/m^2.

The values obtained for the thermal conductivity were assigned to a temperature which was the mean of the junctions of the differential thermocouple, and the absolute temperature of one junction was measured, together with the temperature gradient. The table gives the experimentally determined values of thermal conductivity of paraffin

TABLE

Values of thermal conductivity of paraffin as a function of temperature.

T, °K	λ, mw · cm^{-1} · $degree^{-1}$
295	2.57
264.2	2.73
233.6	2.78
203.4	2.79
173.2	2.72
142.8	2.36
108.3	1.64
83.5	1.37

in the temperature range 83-295°K. The conductivity increases as the temperature drops from 295°K, passes through a maximum in the vicinity of 200°K, and exhibits a sharp fall with further decrease in temperature.

The maximum relative error due to inaccuracy in measuring the geometrical dimensions of the specimen and the temperature gradient in it, does not exceed 1%. The error in determining the power dissipated in the specimen heater is 0.05%. The overall error in conductivity determination does not exceed 3%. Reference [3] gives a value of the conductivity of paraffin at 20°C which differs from that obtained here by 3.5%. Taking into account the experimental error, and the possibility of some difference in specimen composition, this agreement must be considered good.

The method developed may be used for laboratory determination of the thermal conductivity of insulators at low temperatures in cases when high accuracy is required.

NOTATION

β — volume coefficient of thermal expansion; ϵ — emissivity of surface; R — specimen radius; l — specimen length; h — gap between specimen and screen; x, r — cylindrical coordinates of a point on the specimen; T(x, r) — temperature distribution in specimen; T_0 — temperature of lower end of specimen; θ — difference T - T_0; q — thermal flux density; λ — thermal conductivity; ϑ — angle in the $\vec{n}\vec{x}$ plane between the normal $\vec{n}$ and the direction of radiation; $\vec{n}$ — vector normal to specimen surface; $J(\vartheta, x)$ — energy radiated from unit specimen surface per unit time at angle ϑ; σ — Stefan's constant; $\tau(x, r)$ — correction to temperature distribution due to radiative thermal conduction across vacuum chamber; I_0, I_1 and K_1 — Bessel functions of imaginary argument.

REFERENCES

1. H. K. White, Experimental Techniques in Low-Temperature Physics [Russian translation], Fizmatgiz, 1961.
2. V. S. Chirkin, Thermal Conductivity of Industrial Materials [in Russian], Mashgiz, 1962.
3. Handbook of Chemistry and Physics, 37-th Ed., Chemical Rubber Publishing, Cleveland, 1956.

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