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RAPID METHODS FOR DETERMINATION OF THERMAL CONDUCTIVITY COEFFICIENTS OF THERMOELECTRIC MEATERIALS IN SEMICONDUCTOR THERMOBATTERIES

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Methods are developed for absolute and relative determination of the thermal conductivity coefficient of specimens of semiconductor material, with the only measurements required being determination of the relative temperature heads between the external medium and the specimen faces when controlled heat exchange is created at the external surface.

The net temperature-energy efficiency of a semiconductor cooler, especially a multistage one, depends to a significant degree on the correspondence of the semiconductor thermoelectric properties – thermo-emf coefficient, thermal conductivity, and electrical conductivity, used in calculations, to the real properties of the materials in the semiconductor stages.

Refinement of the temperature dependences of material thermoelectric properties at the temperature realized in semiconductor stages is also of great importance for the purpose of correcting data used to perform calculations.

When high efficiency batch methods are used to prepare thermobattery micromodules, or with use of high efficiency semiconductor materials produced by directed crystallization causing significant radial and axial anisotropy, the parameters of individual elements can differ greatly from the standard specifications of the billet from which they were cut. Therefore a necessary stage which must be included in the design of semiconductor coolers is rapid determination of material thermoelectric properties directly in the devices, refinement of their actual temperature dependences, and rejection of specimens whose properties do not correspond to those used in the calculations. The problem of developing high speed methods for determining the thermal conductivity coefficient of large quantities of semiconductor specimens is especially complex.

The major source of error in existing absolute and comparative methods for measuring thermal conductivity of semiconductor specimens is the complexity of precisely determining the thermal flux passing through the specimen and thermal losses from lateral surfaces [1,

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2]. In the methods to be described below for experimental measurement of κ, heat exchange on the lateral specimen surface is not compensated by reference heaters, but is deliberately induced, while monitoring its intensity.

Theory of the Method. The temperature distribution in the semiconductor specimen, the entire surface of which, aside from the thermal contact boundary, is exchanging heat with the external medium, is a solution of the steady state thermal conductivity differential equation

$$\frac{d^2T(x)}{dx^2}-\frac{\alpha p}{\varkappa s}(T(x)-T_m)=0 \tag{1}$$

with boundary conditions:

$$x=0 \quad -\varkappa s\frac{dT(x)}{dx}=Q, \tag{2}$$

$$x=l \quad -\varkappa s\frac{dT(x)}{dx}=\alpha(T(l)-T_m). \tag{2'}$$

The solution of Eq. (1) with boundary conditions (2), (2') has the form

$$T(x)=T_m+\left(\frac{Q}{s}\operatorname{ch}k(l-x)-\alpha(T(l)-T_m)\operatorname{ch}kl\right)(\varkappa k\operatorname{sh}kl)^{-1}, \tag{3}$$

where

$$k=\sqrt{\frac{\alpha p}{\varkappa s}}. \tag{4}$$

Substituting in Eq. (3) the values x = 0 and x = ℓ, we obtain:

$$T(0)-T_m=\left(\frac{Q}{s}\operatorname{ch}kl-\alpha(T(l)-T_m)\right)(\varkappa k\operatorname{sh}kl)^{-1}, \tag{5}$$

$$T(l)-T_m=\left(\frac{Q}{s}-\alpha(T(l)-T_m)\operatorname{ch}kl\right)(\varkappa k\operatorname{sh}kl)^{-1}. \tag{6}$$

It follows from Eq. (6) that:

$$Q=s(T(l)-T_m)(\varkappa k\operatorname{sh}kl+\alpha\operatorname{ch}kl). \tag{7}$$

Substituting Q from Eq. (7) in Eq. (5), we find

$$T(0)-T_m=(T(l)-T_m)(\operatorname{ch}kl(\varkappa k\operatorname{sh}kl+\alpha\operatorname{ch}kl)-\alpha)(\varkappa k\operatorname{sh}kl)^{-1}.$$

After simple transformations we obtain

$$\varphi=\operatorname{ch}kl+\frac{\alpha}{\varkappa k}\operatorname{sh}kl, \tag{8}$$

where

$$\varphi=\frac{T(0)-T_m}{T(l)-T_m}. \tag{9}$$

Thus, the theory of the method itself eliminates the need for experimental determination of the thermal flux Q.

By using Eq. (4) to transform the expression $\alpha/\varkappa k$ to the form

$$\frac{\alpha}{\varkappa}\sqrt{\frac{\varkappa s}{\alpha p}}=\frac{D}{4l}kl,$$

we can transform Eq. (8):

$$\varphi=\operatorname{ch}kl+\frac{D}{4l}kl\operatorname{sh}kl. \tag{10}$$

Equation (10) establishes an implicit relationship between the thermal conductivity coefficient of the semiconductor specimen, the intensity of heat exchange on the surface

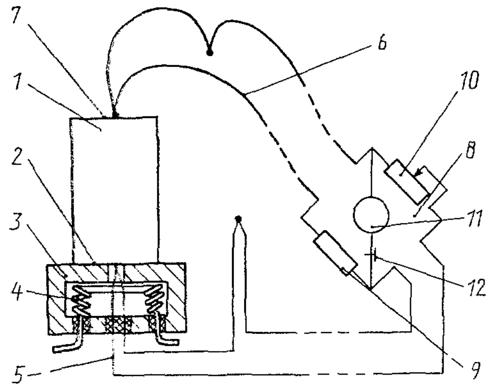

Fig. 1. Experimental apparatus for determination of thermal conductivity coefficient by the absolute method: 1, specimen; 2, thermal contact boundary; 3, body; 4, heater; 5, 6, thermocouples; 7, free boundary; 8, resistive bridge; 9, reference resistor; 10, precision variable resistor; 11, null galvanometer; 12, pushbutton.

facing the external medium, the temperatures of the specimen faces and the external medium, and the geometric dimensions of the specimen.

Absolute and relative variants of the κ measurement technique were developed, based on the physical approach described above.

In the absolute method the thermal conductivity coefficient is calculated from the expression

$$\varkappa_{sp}=\frac{4l^2}{Dy^2}\alpha, \tag{11}$$

where $y = kl$ is a root of transcendental equation (10).

During measurements (Fig. 1) a temperature head is created by simultaneous action of the gradient heater and heat exchange with an air flow on the lateral surface and free face of the specimen. The heat liberation coefficient between the specimen surface and the air flow is measured by the methods described in [3, 4]. The resistive bridge is balanced by changing the precision variable resistor, and Eq. (9) used to find the value of φ; when thermocouples with identical thermo-emf coefficients are used the resistance ratio yields the value of φ.

To estimate the uncertainty of the κ_{sp} determination from Eq. (11) we obtain

$$\frac{\Delta\varkappa_{sp}}{\varkappa_{sp}}=\pm\sqrt{4\left(\frac{\Delta l}{l}\right)^2+\left(\frac{\Delta D}{D}\right)^2+\left(\frac{\Delta\alpha}{\alpha}\right)^2+4\left(\frac{\Delta y}{y}\right)^2}.$$

The total relative uncertainty in measurement of the geometric dimensions of individual specimens comprises 0.3%, while the same value for semiconductor stages in regular production thermobatteries is 1%.

To determine the uncertainty y for various values of φ we find the derivative $dy/d\varphi$ from Eq. (10)

$$\frac{dy}{d\varphi}=\left(\left(1+\frac{D}{4l}\right)\operatorname{sh}y+\frac{D}{4l}y\operatorname{ch}y\right)^{-1}.$$

Hence we easily obtain

$$\frac{dy}{y}=F(y(\varphi))\frac{d\varphi}{\varphi},$$

where

$$F(y(\varphi))=\left(\left(1+\frac{D}{4l}\right)\operatorname{sh}y+\frac{D}{4l}y\operatorname{ch}y\right)^{-1}\frac{\varphi}{y}.$$

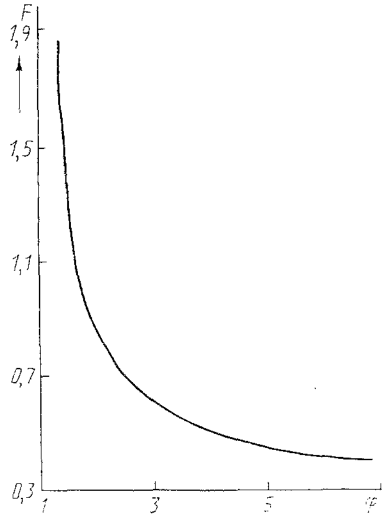

Fig. 2. Dependence on φ of function for determining relative uncertainty of parameter y.

As is evident from Fig. 2, to insure temperature heads across the specimen corresponding to values $\varphi > 1, 2$, the quantity $F(\varphi) < 1$; thus, the relative uncertainty in y determination with change in φ by balancing thermocouple signals with the resistive bridge is less than 1%.

In the final outcome the uncertainty in κ_{sp} determination is determined mainly by the uncertainty in determination of the heat liberation coefficient, which can be reduced to 3-5% by performing the studies in an air flow with a stabilized flow rate.

In the comparative measurement method (Fig. 3) the volume of experimental α measurements is eliminated, and we perform only one additional measurement of the temperature difference between the free surface of a reference specimen and the external medium for identical temperatures of the thermal contact surfaces of the reference specimen and the specimen under study and identical heat exchange coefficients with the external medium.

In this case the expression for calculating κ_{sp} has the form:

$$\kappa_{sp} = \frac{D_{ref}}{D_{sp}} \left(\frac{l_{sp}}{l_{ref}} \right)^2 \left(\frac{y_{ref}}{y_{sp}} \right)^2 \varkappa_{ref}. \tag{12}$$

The relative uncertainty of κ_{sp} determination by the relative method is equal to

$$\frac{\Delta\varkappa_{sp}}{\varkappa_{sp}} = \pm \sqrt{2 \left(\frac{\Delta D}{D} \right)^2 + 8 \left(\frac{\Delta l}{l} \right)^2 + 8 \left(\frac{\Delta y}{y} \right)^2 + \left(\frac{\Delta\varkappa_{ref}}{\varkappa_{ref}} \right)^2}. \tag{12}$$

Analysis shows that the uncertainty in measuring the thermal conductivity coefficient with Eq. (12) for single measurements does not exceed 1-2%, with a corresponding value of 3-4% for serial measurements.

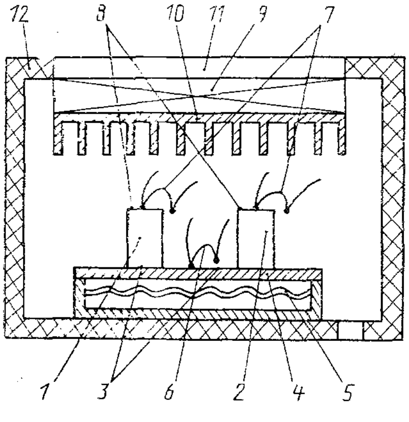

Fig. 3. Experimental apparatus for determination of thermal conductivity coefficient by the relative method: 1, reference specimen; 2, specimen under study; 3, thermal contact face; 4, isothermal base; 5, gradient heater; 6, 7, thermocouples; 8, free surface; 9, thermobattery; 10, heat radiator system; 11, heat exchanger; 12, heat shielding.

These methods for measuring the thermal conductivity coefficient of semiconductor specimens can be used in industrial conditions for rapid determination of the thermal conductivity coefficients of large numbers of semiconductor stages directly in the process of thermobattery construction.

NOTATION

T, temperature, K; κ, thermal conductivity coefficient, $W \cdot m^{-1} \cdot K^{-1}$; α, heat liberation coefficient, $W \cdot m^{-2} \cdot K^{-1}$; Q, thermal flux, W; ℓ, length, m; D, diameter, m; $p = aD$, perimeter ($a = 4$ for square sections, $a = \pi$ for round sections); $s = bD^2$, area, m^2; ($b = 1$ for square sections, $b = \pi/4$ for round sections); x, coordinate, m. Subscripts: m, medium; sp, specimen; ref, reference.

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MEASURED SPECIFIC HEATS OF HEXAN-1-OL AND 3-METHYL-2-BUTANOL OVER WIDE TEMPERATURE RANGES

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The specific heat c_p has been measured for hexan-1-ol and 3-methyl-2-butanol at 227-363 and 218-371 K correspondingly. The measurements for the first are compared with data from other sources.

Various industrial tasks can be handled more efficiently from reliable data on the specific heats of important liquids such as n-alcohols and iso-alcohols, which can also provide more economical organization in the chemical, medical, and perfumery industries.

Table 1 collects the measurements on the specific heat of hexan-1-ol in the liquid state at or near atmospheric pressure. Below 290 K, the experimental c_p in [1] are lower than those in [2] by from 2.3 to 5.6%. Above 290 K, the [3-11] data are in agreement within the over-all error (±2.4%) of the independent measurements [4, 5].

There are no published c_p for liquid isoamyl alcohol (3-methyl-2-butanol).

We have measured c_p for hexan-1-ol at 227-363 K and for 3-methyl-2-butanol between 218 and 371 K for the liquids in equilibrium with the saturated vapor. The substances were of 99.9% purity. The c_p were measured by stepswise heating in an adiabatic calorimeter (temperature step about 4-5 K), which was made by the Khabarovski branch of the All-Union Technical Physics and Electronics Research Institute. The apparatus and methods have been described in detail [12, 13].

One determines c_p from a known mass of material and known heat equivalent for the empty calorimeter by measuring the amount of heat supplied to the calorimeter together with the

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