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CONTACTLESS METHOD OF DETERMINING THE COEFFICIENT OF THERMAL DIFFUSIVITY OF LOCAL DOMAINS OF SURFACE LAYERS AND THIN FILMS

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A method is proposed for measuring the coefficient of thermal diffusivity of microsections of surface layers and thin films. The coefficient of thermal diffusivity is calculated from the time dependence of the heat flux emitted by the heated surface.

The possibility of determining the physical parameters characterizing micron regions of surface layers of bulk specimens and thin films is important in the miniaturization of technical apparatus.

X-ray spectrum electron-probe microanalysis [1] is extensively used at this time. In particular, this method is used to investigate semiconductor materials and thermoelectric substances, which permits obtaining data about the microinhomogeneity of the substance, the composition of the shallow phases, the intermediate layers, the behavior of the doping impurities, etc. [2]. Knowledge of only the results of an x-ray spectrum microanalysis is insufficient for an estimation of the role of the microinhomogeneities, the shallow phases, and the intermediate layers in semiconductor instruments. Knowledge of the physical parameters of the microdomains under investigation, particularly the thermophysical properties, is also important.

We used an electron beam x-ray microanalyzer of MS-46 type in combination with a high sensitivity infrared radiation detector to determine the coefficient of thermal diffusivity of surface or film microsections. Since the diameter of the electron beam of instruments of the type mentioned equals approximately 1 μm, the measured values of the coefficient of thermal diffusivity will characterize a domain several microns in size. The crux of the method is the following.

A cylindrical section of radius a and thickness l (Fig. 1) of the surface of the specimen 3 under investigation is exposed to the axisymmetric electron beam 1 whose density is normally distributed.

Heat will be liberated at the site of electron beam incidence on the surface being investigated, part of which will be dissipated in the substance and part of which will be radiated by the surface into surrounding space. It may be considered that the quantity of energy being radiated is negligible compared to the quantity which is dissipated within the substance.

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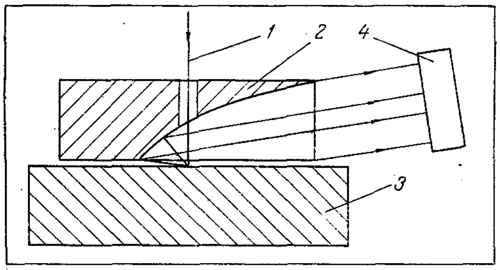

Fig. 1. Diagram of an apparatus for contactless determination of $\varkappa$.

The heat radiated by the surface by using a parabolic mirror 2 at whose focus is the center of the electron beam and a gilded cone is delivered to the infrared radiation detector 4, whose signal is amplified by a special amplifier.

Let us consider the case when the time to heat the surface is so great that a stationary temperature field $T = T(r, z)$ succeeds in being built up in the body. At the time $t = 0$ let the heating cease and cooling of the body start. The dependence of the temperature on the coordinates is evidently determined by solving the equation

$$\frac{\partial^2 T}{\partial r^2} + \frac{1}{r}\cdot\frac{\partial T}{\partial r} + \frac{\partial^2 T}{\partial z^2} = \frac{1}{\varkappa}\cdot\frac{\partial T}{\partial t} \tag{1}$$

under the following conditions

$$T = T_0 \quad \text{for} \quad r = a \text{ and } z = l, \tag{2}$$

$$T = T(r,\ z) \quad \text{for} \quad t = 0. \tag{3}$$

It should be noted that the temperature is independent of the polar angle because of axial symmetry.

The function $T(r, z)$ describing the stationary temperature field distribution is evidently the solution of the equation

$$\frac{\partial^2 T}{\partial r^2} + \frac{1}{r}\cdot\frac{\partial T}{\partial r} + \frac{\partial^2 T}{\partial z^2} = 0 \tag{4}$$

under the conditions $T = T_0$ for $r = a$, $z = l$,

$$-k\left.\frac{\partial T}{\partial z}\right|_{z=0} = \frac{q_0}{2\pi\sigma^2}\exp\left(-\frac{r^2}{2\sigma^2}\right). \tag{5}$$

The right-hand member in (5) is proportional to the electron-beam density and can be written in that form under the condition that

$$\sigma \ll a. \tag{6}$$

Taking into account that the size of an electron beam in an MS-46 x-ray microanalyzer is around 1 μm, it can be considered that (6) is practically always satisfied.

The solution of (5) has the form [3]

$$T(r,\ z) - T_0 = \frac{q_0}{2ak}\sum_{n=1}^{n=\infty} I_0\left(\lambda_n\frac{r}{a}\right)\frac{\operatorname{sh}\left[\lambda_n\dfrac{l-z}{a}\right]}{\operatorname{ch}\left[\lambda_n\dfrac{l}{a}\right]}\exp\left[-\left(\frac{\lambda_n}{a}\right)^2\frac{\sigma^2}{2}\right], \tag{7}$$

where λ_n is a root of the equation

$$I_0(\lambda_n) = 0. \tag{8}$$

Equation (7) describes the temperature field at the time $t = 0$ and is an initial condition for (1).

Solving (1) in combination with the conditions (2) and (3), we obtain the solution in the form of a series expansion in the eigenfunctions [4]

$$T-T_0=\sum_{m=1}^{m=\infty}\sum_{n=1}^{n=\infty}A_{mn}I_0\left(\frac{\lambda_n}{a}r\right)\sin\frac{m\pi}{2l}(l-z)\exp\left[-\left(\frac{\lambda_n^2}{a^2}+\frac{m^2\pi^2}{4l^2}\right)\varkappa t\right]. \tag{9}$$

Since the series (9) and (7) should agree at the initial instant, by comparing (9) and (7) we obtain for A_{mn} (taking account of the orthogonality of the trigonometric functions in the segment $\pm l$)

$$A_{mn}=\frac{q_0\exp\left[-\left(\frac{\lambda_n}{a}\right)^2\frac{\sigma^2}{2}\right]}{alk\,\mathrm{ch}\left(\frac{\lambda_n}{a}l\right)}\frac{\left[\frac{\lambda_n}{a}\,\mathrm{ch}\left(\frac{\lambda_n}{a}l\right)\sin\frac{m\pi}{2}-\frac{m\pi}{2l}\,\mathrm{sh}\left(\frac{\lambda_n}{a}l\right)\cos\frac{m\pi}{2}\right]}{\left(\frac{\lambda_n}{a}\right)^2+\left(\frac{m\pi}{2l}\right)^2}. \tag{10}$$

Therefore, (9) in combination with (10) indeed yields the temperature distribution for the times $t \geq 0$. Since the total heat flux emitted by a heated surface is measured in our case, it must then be determined. We have for the heat flux Q

$$dQ=\varepsilon\sigma_0(T^4-T_0^4)\,2\pi r dr. \tag{11}$$

Considering that heating of a surface by an electron beam is slight and taking into account that $Q \sim (T - T_0)$ for $(T - T_0)/T_0 \ll 1$, we obtain from (9), (10) and (11) for $z = 0$

$$Q=\frac{8\pi\varepsilon\sigma_0T_0^3q_0}{kl}\sum_{n=1}^{n=\infty}I_1(\lambda_n)\exp\left[-\left(\frac{\lambda_n}{a}\right)^2\left(\frac{\sigma^2}{2}+\varkappa t\right)\right]\sum_{m=1}^{m=\infty}\frac{\exp\left[-\left(\frac{m\pi}{2l}\right)^2\varkappa t\right]}{\left(\frac{\lambda_n}{a}\right)^2+\left(\frac{m\pi}{2l}\right)^2}\sin^2\frac{m\pi}{2}, \tag{12}$$

which describes the time dependence of the total energy flux radiated by the surface. It is seen from (12) that by knowing the logarithm of Q as a function of the time, it is always possible to compute the coefficient of thermal diffusivity $\varkappa$ if the dimensions a and l of the specimen are known.

Let us examine several particular cases.

1. $l \ll a$. In this case we have from (12) for sufficiently large t

$$Q=\frac{8\pi\varepsilon\sigma_0T_0^3q_0}{kl}I_1(\lambda_1)\exp\left[-\left(\frac{\lambda_1}{a}\right)^2\frac{\sigma^2}{2}\right]\frac{4l^2}{\pi^2}\exp\left[-\frac{\pi^2}{4l^2}\varkappa t\right], \tag{13}$$

since $\lambda_1/a \ll \pi/2l$.

In this case the proposed experimental method may, in principle, evidently be applied to determine the quantity $\varkappa$ of thin films.

2. $l \gg a$. The quantity $\varkappa$ to be measured will evidently characterize the near-surface layer of dimension $\sim\sigma$ of the material being investigated in this case.

In this case, we obtain the following from (12) by setting $a \ll l$:

$$Q=\frac{8\pi\varepsilon\sigma_0T_0^3q_0}{kl.}I_1(\lambda_1)\exp\left[-\left(\frac{\lambda_1}{a}\right)^2\left(\frac{\sigma^2}{2}+\varkappa t\right)\right]\left(\frac{a}{\lambda_1}\right)^2. \tag{14}$$

It should be noted that another method exists for measuring the coefficient of thermal diffusivity, which has some advantages over the method of determining $\varkappa$ by measuring the cooling tempo. The second method is based on measuring the quantity of heat being radiated by the heated surface during some time span. The requirements on the inertia of the transducer receiving the thermal radiation are hence diminished.

Setting $t = 0$ in (12), we obtain an equation describing the heat flux Q_0 being radiated by a heated surface in the stationary mode:

$$Q_0=\frac{8\pi\varepsilon\sigma_0T_0^3q_0}{kl}\sum_{n=1}^{n=\infty}I_1(\lambda_n)\exp\left[-\left(\frac{\lambda_n^2}{a}\right)\frac{\sigma^2}{2}\right]\sum_{m=1}^{m=\infty}\frac{\sin^2\frac{m\pi}{2}}{\left(\frac{\lambda_n}{a}\right)^2+\left(\frac{m\pi}{2l}\right)^2}. \tag{15}$$

From (15), the total quantity of heat Q_{10} during a time Δt will be

$$Q_{10}=Q_0\Delta t. \tag{16}$$

From (12) we will have for the total quantity of heat Q_{20} being radiated after cessation of the heating,

$$Q_{20}=\int_{0}^{\infty} Q\,dt=\frac{8\pi\varepsilon\sigma_0 T_0^3 q_0}{kl}\,\frac{1}{\varkappa}\sum_{n=1}^{n=\infty} I_1(\lambda_n)\exp\left[-\left(\frac{\lambda_n}{a}\right)^2\frac{\sigma^2}{2}\right]\sum_{m=1}^{m=\infty}\frac{\sin^2\frac{m\pi}{2}}{\left[\left(\frac{\lambda_n}{a}\right)^2+\left(\frac{m\pi}{2l}\right)^2\right]^2}. \quad (17)$$

From (16) and (17) we obtain

$$\varkappa=\frac{Q_{10}}{Q_{20}}\times\frac{\sum_{n=1}^{n=\infty} I_1(\lambda_n)\exp\left[-\left(\frac{\lambda_n}{a}\right)^2\frac{\sigma^2}{2}\right]\sum_{m=1}^{m=\infty}\frac{\sin^2\frac{m\pi}{2}}{\left[\left(\frac{\lambda_n}{a}\right)^2+\left(\frac{m\pi}{2l}\right)^2\right]^2}}{\sum_{n=1}^{n=\infty} I_1(\lambda_n)\exp\left[-\left(\frac{\lambda_n}{a}\right)^2\frac{\sigma^2}{2}\right]\sum_{m=1}^{m=\infty}\frac{\sin^2\frac{m\pi}{2}}{\left(\frac{\lambda_n}{a}\right)^2+\left(\frac{m\pi}{2l}\right)^2}} \quad (18)$$

Therefore, by using the measured quantities of the total radiated thermal energy from the heated surface the magnitude of the coefficient of thermal diffusivity $\varkappa$ can be determined.

A simple analysis shows that the series in (18) converge sufficiently rapidly. In practice, it is sufficient to sum the series in (18) up to $n \approx 10$ and $m \approx 10$ by using an electronic computer.

Computations performed show that the energy flux emitted by a heated surface is approximately 10^{-10} W for heating of a surface microsection 10° above room temperature.

To record such low energy fluxes in the infrared range, an infrared radiation detector based on mercury-doped germanium can be used. The voltage sensitivity of such a photodetector is 10^4 V/W at the temperature of liquid helium and the threshold sensitivity to the radiation of an absolutely black body at T = 400°K is $\sim 11^{-11}$ W/cm $\cdot$ $Hz^{1/2}$. In the presence of an appropriate photodetector signal amplifier both the cooling tempo and the total quantity of energy radiated from a heated surface microsection can be recorded experimentally.

NOTATION

r, distance between any point of the body to the cylindrical specimen; z, cylinder axis with origin on the surface being heated and positive direction along the normal to the surface into the body; T, temperature on the surfaces $r = a$ and $z = l$; $\varkappa$, coefficient of thermal diffusivity; k, coefficient of thermal conductivity; σ, normal distribution law parameter for the electron-beam density over the section; q_0, total quantity of heat being liberated per unit time on the specimen surface; $I_0(\lambda_n)$, zero-order Bessel function of the first kind; ε, surface emissivity; σ_0, Stefan–Boltzmann constant; $I_1(\lambda_n)$, first-order Bessel function of the first kind; n, m, arbitrary integers.

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