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Two-beam photoacoustic phase measurement of the thermal diffusivity of solids

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A simple method is demonstrated for obtaining the thermal diffusivity of solids, by measuring the phase lag between a front and rear illumination, at a single chopping frequency. The method is tested using some semiconductor and glass samples.

I. INTRODUCTION

The quantity that measures the rate of diffusion of heat in a material is called the thermal diffusivity α . Apart from its own intrinsic importance, its determination gives the value of the thermal conductivity k , if the density ρ and the thermal capacity at constant pressure are known, since

$$\alpha = k / \rho c. \quad (1)$$

The experimental methods for determining the thermal diffusivity are of two types, depending on whether the measured heat flow is transient or periodic.^{1,2} The latter method was introduced as early as 1861 by Ångström, and consists basically of a rod that is periodically heated at one point, and whose temperature oscillation is measured at another point. The phase lag between the thermal oscillation at any two points gives a precise determination of α , as was done by Hirschman *et al.*³ They measured the phase of the nonilluminated rear-end surface-temperature oscillation in relation to the phase of the square-wave radiation incident on a thin disc.

In the photoacoustic effect, a sample enclosed in a cell with air is heated by a chopped light beam, and there is a periodic heat transfer between the sample and the air. The signal, generated by the resultant pressure oscillation in the air and detected by a microphone in the cell, is proportional to the temperature oscillation on the sample's surface.⁴ The photoacoustic effect then constitutes a suitable technique for measuring the thermal diffusivity by the phase-lag method.

The importance of simple thermal diffusivity measurements can be seen in its application to semiconductors. Power dissipation in microelectronic and optoelectronic devices is an important mechanism in limiting device performance. For example, in semiconductor diode lasers, heat generated in the active layer (eg., GaAs) must pass through a substantial thickness of a confining layer of higher band gap and lower refractive index (eg., Ga_{1-x}Al_xAs) to reach the heat sink. The room-temperature value of the thermal diffusivity of the materials used in these devices is therefore an important parameter in their optimization.⁵

II. DIFFUSIVITY MEASUREMENTS BY PHOTOACOUSTIC EFFECT

Various methods have been developed to measure the thermal diffusivity by means of the photoacoustic effect. Ad-

ams and Kirkbright⁶ calculated α for various samples (coated with a very thin layer of black enamel to ensure surface absorption) by plotting the phase ψ of the photoacoustic signal, obtained from rear-surface illumination, vs $\sqrt{\omega}$, where ω is the chopping angular frequency. Their supposition that $\psi = la_s$, where $a_s = (\omega/2\alpha)^{1/2}$ and l is the sample's thickness, is an approximation for the thermally thick case (as $la_s \gg 1$).

An equivalent procedure^{7,8} is to measure, instead of the phase, the attenuation of the magnitude of the thermal oscillation as a function of $\sqrt{\omega}$, again for the case of rear-surface illumination. The frequency ω for which the slope of the attenuation curve diverges from the value of -3 is typical of thermally thick samples and indicates an $la_s = 1$ point, at which extraction of the thermal diffusivity α may be made.

Another alternative to the Ångström method is to vary the distance x_0 between the point where the heat is generated and one point at which the thermal oscillation is measured. Cesar *et al.*⁹ adapted this method to photoacoustics, by varying the position of a laterally incident laser beam on a transparent sample. The plot of both the photoacoustic phase and magnitude versus the distance x_0 provided α by a data fit to expressions with, respectively, a linear and an exponential dependence on $-a_s x_0$.

For samples not necessarily thermally thick, but having a large optical-absorption coefficient β (which means that the conditions $\beta l \gg 1$ and $\beta \gg a_s$ are valid), an attractive and simple method has been introduced by Yasa and Amer.¹⁰ They have measured the attenuation of the rear-surface illumination signal (S_R) relative to the front-surface illumination signal (S_F). Using the appropriate formula, they were able to obtain from the ratio S_F/S_R . The method we propose in what follows uses the same experimental setup, measuring, however, the relative phase-lag $\Delta\psi = \psi_F - \psi_R$, instead of the signal-magnitude ratio.

III. THEORY

The theoretical expression for the ratio S_F/S_R and for the phase-lag $\Delta\psi$ can be obtained from the Rosencwaig-Gersho theory,⁴ applied to both front-and rear-surface illuminations.⁸ The temperature amplitude on the sample-air interface, which is proportional to the photoacoustic signal,

is given, for these cases, by

$$\theta_F = \frac{I_F}{k_s \sigma_s} \frac{\cosh(l\sigma_s)}{\sinh(l\sigma_s)}, \quad (2)$$

$$\theta_R = \frac{I_R}{k_s \sigma_s} \frac{1}{\sinh(l\sigma_s)}, \quad (3)$$

where $\sigma_s = (1 + j)a_s$ and I_F and I_R are the absorbed incident light intensities at the front and rear sides, respectively. In arriving at Eqs. (2) and (3) we have assumed that the sample is optically opaque to the incident light and that the heat flux into the surrounding air is negligible. These approximations correspond to the actual experimental situation and represent no limitation of our method. The ratio of the magnitudes and the phase difference between the front and rear signals are then

$$\frac{S_F}{S_R} = \frac{I_F}{I_R} [\cosh^2(la_s) - \sin^2(la_s)]^{1/2} \quad (4)$$

and

$$\tan(\Delta\psi) = \tanh(la_s) \tan(la_s). \quad (5)$$

Equation (4) depends explicitly on the ratio I_F/I_R . This means that in the signal-magnitude ratio measurement, precise power monitoring of each beam and identical surface conditions on each side of our samples are needed. Yasa and Amer,¹⁰ using Eq. (4) calculated the thermal diffusivity from the slope of the value of the curve S_F/S_R for several modulation frequencies. This is equivalent to performing an average for each frequency. In contrast, Eq. (5) exhibits no explicit dependence on the absorbed power and surface conditions. This implies that in the phase-lag method a single-chopping frequency measurement is sufficient to derive the thermal diffusivity. Furthermore, the independence of the phase-lag method to power calibrations and surface conditions renders it as a more precise technique than the magnitude ratio method. The usefulness of phase measurements for measuring the thermal diffusivity has already been recognized in the past by several authors. In fact, Eq. (5) was known to Hirschman *et al.* since the early sixties. They did not use it, however, because of the difficulty they had in the thermocouple measurement of the temperature on the front surface. Instead, they compared (as Adams and Kirkbright⁶ did) the phase of the chopping frequency of the incident radiation, using a different and more complicated formula.²

IV. RESULTS AND DISCUSSION

To demonstrate the usefulness, generality, and accuracy of the two-beam phase-lag method outlined above we have conducted the thermal diffusivity measurement of several semiconducting samples and of a highly transparent sample, namely, a BK-7 glass sample. The arrangement of our two-beam experiment is schematically shown in Fig. 1. A chopped argon ion laser beam is divided by a beamsplitter, and each beam is directed to opposite sides of the photoacoustic cell. The plane-shaped sample is fixed with silicon gel to a brass ring that has a 4-mm-diam hole through which the rear-side laser beam is incident. This corresponds to an air backing. Some of the heat generated in the sample, resulting from the light absorption, is lost to the brass ring, but this

FIG. 1. Experimental setup for the two-beam photoacoustic measurements of the thermal diffusivity.

is of little effect considering the short thermal-diffusion length of the sample. In order to check the reproducibility of our results, the phase measurements of our samples were carried out at several modulation frequencies in the interval between 25 and 160 Hz. The semiconducting samples used were: (1) Ge(100) *n*-type, 784 μm thick; (2) Si(111), *p*-type, 345 μm thick; (3) GaAs(100), *n*-type, 568 μm thick; (4) GaSb, 420 μm thick; (5) InP(100), *n*-type 442 μm thick; (6) InAs, 550 μm thick. For these semiconductors the large optical-absorption coefficient (i.e., $\beta > 10^4 \text{ cm}^{-1}$) for the argon laser light ensures us the condition of optical opaqueness implicit in Eqs. (2)–(5). In the case of the BK-7 glass sample (320 μm thick) the condition of optical opaqueness, implicit in Eq. (5), was achieved in two different ways. In one case, the glass sample was coated on both sides with a thin Cu film, 3800 Å

TABLE I. Comparison of the thermal diffusivity values obtained from the two-beam phase-lag method with the values quoted in the literature.

Material	Measured (cm^2/s)	Standard deviation	Literature values of α
Ge	0.35	0.01	0.346 ^a
Si	0.94	0.03	0.880 ^a
GaAs	0.24	0.01	0.21–0.26 ^a
GaSb	0.24	0.01	0.24 ^b
InP	0.45	0.02	0.46 ^b
InAs	0.22	0.01	0.19 ^b
BK-7	0.0058	0.0006	0.0053 ^c
(Cu-coated)			
BK-7	0.0055	0.0004	0.0053 ^c
(Al foil)			

^a Reference 1.

^b Reference 11.

^c Reference 12.

FIG. 2. Plot of $\Delta\psi$ vs la_s [Eq. (5)] showing the experimental values obtained for Ge and Si using the observed values of α .

thick. In the other case, a thin circular Al foil (20 μm thick) of 3-mm diameter was glued to both sides of the BK-7 sample using a thin layer of a thermally conducting grease (Thermal Compound No. 120-8, Wakefield Eng. Inc.). This second way of ensuring the optical opaqueness was a necessary applicability test of the technique. For some samples, like polymers and biological samples, the deposition of an absorbing thin film is either extremely difficult or undesirable because the deposition process may change the sample properties. Furthermore, the use of black paints, in the case of samples with small thermal diffusivity may also inhibit the measurement of the sample thermal diffusivity. We have tested these two techniques for the case of a biological sample, namely, the external envelope (pericarp) of a corn kernel. The thin-film deposition technique was certainly not adequate since the dehydration of our sample during the deposition process was changing its properties. As to the use of a black spray paint, the problem becomes that of a two-layer system for which it is wrong to assume that each layer contributes separately to the phase shift. The use of the thin Al foil glued to the transparent sample proved to be an extremely simple and reliable method.

In Table I, we summarize the results of our measurements of the thermal diffusivity and compare them with the literature values. In Fig. 2, we show the dependence of the phase-lag $\Delta\psi$ as a function of $la_s = l(\pi f/\alpha_s)^{1/2}$, as given by Eq. (5). Also shown in Fig. 2 are the experimental values for our Ge and Si samples, using the observed values given in Table I. It follows from this plot that our phase-lag method is valid for all values of lag, i.e., for both thermally thick

($la_s \gg 1$) and thermally thin ($la_s \lesssim 1$) samples. In contrast, the Adams and Kirkbright⁶ method is valid only for the case of thermally thick samples ($la_s > 1.4$), when the slope of $\Delta\psi$ with respect to a is one (i.e., dashed line on Fig. 2). The very good agreement between our observed values of the thermal diffusivity and the literature values shows that the proposed two-beam photoacoustic phase measurement is a simple, fast, and accurate method for obtaining the thermal diffusivity. Its advantages over the conventional photoacoustic techniques is that the thermal diffusivity is obtained from a single-chopping frequency measurement, it is not limited to thermally thick samples, and does not depend on the beam power calibrations and the sample surface conditions.

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