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Thermal Diffusivity Measurements of Foil-Shaped Materials by Vectorial Analysis Using an AC Calorimeter[1]

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Measurements of thermal diffusivity of foil-shaped materials have been carried out using a photoirradiation-type AC calorimeter at room temperature. In this method the frequency effect, which is caused by heat loss from the sample to the environment, is readily detected in measurements of both amplitude and phase components of the AC temperature signal. Even though the chopping frequency is appropriate, the two diffusivities calculated from these two components differ from each other. Moreover, the difference between the two values increases when the chopping frequency increases. Simple vectorial calculation with the two components—one from the amplitude and the other from the phase—permits the frequency effect to be determined. The calculated result is the geometric average of the two diffusivities. This analytic method was tested with diamond film and SUS-304 foil. From these we confirmed that the vectorial analytic method gives similar diffusivity values for different frequencies indicating its reliability.

KEY WORDS: AC calorimeter; frequency effects; thermal diffusivity; vectorial analysis.

1. INTRODUCTION

The nature of heat transport in thin film materials has received much attention in recent years. One technique for the measurement of thermal diffusivity of thin specimens, the photoirradiation-type AC calorimeter, was

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developed by Hatta [1] in 1985. This method is convenient for determining the thermal diffusivity in a direction parallel to the surface of foil-shaped specimens. In this method, AC light with uniform intensity is masked so that it impinges on a limited portion of the sample surface. The decay of the AC temperature at a thermocouple located in the masked region is measured as a function of distance between the thermocouple and the edge of the mask. The thermal diffusivity can be obtained from the logarithm of the amplitude or the phase shift of the AC temperature waves. In their serial reports [2–5], several error effects were systematically investigated, i.e., the effect of thermocouple attachment, dimension of sample, heat loss, etc. But they used the amplitude component only as the AC temperature signal. However, the measured value of the thermal diffusivity from their method may deviate significantly from the correct value because of heat loss from sample. This effect is readily detected from the change of thermal diffusiviy with chopping frequency.

In the present paper, we suggest another method using a photoirradiation AC calorimeter to determine the thermal diffusivity of thin samples. Taking the amplitude and phase components of the AC temperature signal simultaneously, it is possible to estimate the heat loss effects with a simple vectorial calculation.

2. THEORY

Consider an infinite sample of thickness d which is partially shadowed from irradiation of the light source by a mask (shown in Fig. 1). The AC heat is $Q(t) = Q\exp(i\omega t)$, where Q is the amplitude of heat flux and ω is

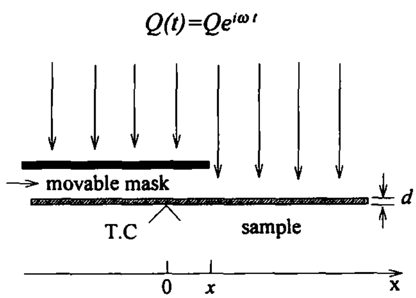

Fig. 1. Cross-section schematic view of the photoirradiation-type AC calorimeter. The movable mask is controlled by a micro-scanner.

the angular frequency. In heat transfer theory, one defines the thermal wave number, k, as

$$k = \sqrt{\frac{\pi f}{\alpha}} \tag{1}$$

where f is the chopping frequency ($\omega = 2\pi f$) and α is the thermal diffusivity. If the thickness of the sample is sufficiently less than the thermal diffusion length, then this system can be treated as a one-dimensional problem. When AC thermal energy is applied, heat waves propagate along the direction of x in the shadowed region. To find the AC temperature deviation from ambient temperature T as a function of position, x, and time, t, one must solve the heat conduction equation.

$$cd\frac{\partial T}{\partial t} + \delta T - \kappa d\frac{\partial^2 T}{\partial x^2} = Q(x, t) \tag{2}$$

with

$$Q(x, t) = Qe^{i\omega t} \qquad \text{for} \quad x \leqslant 0 \tag{3a}$$

and

$$= 0 \qquad \text{for} \quad x > 0 \tag{3b}$$

where c is the heat capacity per volume, κ is the thermal conductivity satisfying the relation $\alpha = \kappa/c$, and δ is the thermal conductance per unit area between the sample and a thermal bath with constant temperature. The solution of Eq. (2) is

$$T(x) = \frac{Q}{2(i\omega cd)} \exp\left[\left\{-\sqrt{\frac{i\omega cd + \delta}{\kappa d}}\right\} x\right] \qquad \text{for} \quad x > 0 \tag{4}$$

By defining the external relaxation time

$$\tau_e = cd/\delta \tag{5}$$

Equation (4) can be rewritten as

$$T(x) = \frac{Q}{i2\omega cd(1 - i(1/\omega\tau_e))} \exp\left[-\left\{\sqrt{i\frac{\omega}{\alpha}\left(1 - i\frac{1}{\omega\tau_e}\right)}\right\} x\right] \tag{6}$$

If the condition of

$$1/\omega\tau_e \ll 1 \tag{7}$$

is satisfied, then Eq. (6) can be approximated by

$$T(x) = \frac{Q}{2\omega cd} \exp\left[-kx - i\left(kx + \frac{\pi}{2}\right)\right] \tag{8}$$

However, if Eq. (7) is not satisfied, as Ångström's first employment [6], Eq. (6) can be rewritten in the following vector form:

$$\vec{T}(r, \theta) = \frac{Q}{\omega cd\sqrt{1+\beta^2}} \exp[-k_r x - i(k_\theta x - \varphi)] \tag{9}$$

where

$$k_r = \sqrt{\frac{\omega}{2\alpha_r}} \equiv \sqrt{\frac{\omega}{2\alpha}(\sqrt{1+\beta^2}+\beta)} \tag{10}$$

$$k_\theta = \sqrt{\frac{\omega}{2\alpha_\theta}} \equiv \sqrt{\frac{\omega}{2\alpha}(\sqrt{1+\beta^2}-\beta)} \tag{11}$$

and

$$\beta = \frac{1}{\omega\tau_e}, \qquad \varphi = \tan^{-1}\left(\frac{1}{\beta}\right) \tag{12}$$

The thermal diffusivity and external relaxation time of a thin sample can be calculated from the experimentally determined k_r and k_θ using Eq. (1) to define α_r and α_θ and

$$\alpha = \frac{\pi f}{k_r k_\theta} = \sqrt{\alpha_r \alpha_\theta} \tag{13}$$

and

$$\tau_e = \frac{1}{\alpha(k_r^2 - k_\theta^2)} \tag{14}$$

This means that both the amplitude and the phase components of the AC temperature must be measured to determine the thermal diffusivity of thin specimens.

3. EXPERIMENTS

We used an expanded Ar-ion laser beam as the energy source, and the system was equipped with a lock-in amplifier (EG&G 5302), microscanner, and digital voltmeter. All were controlled by an IBM-PC. Two samples were chosen: 50-μm-thick SUS-304 (made by Goodfellow Corp.), a low-thermal diffusivity metal alloy, and a diamond film with a high thermal diffusivity. The diamond film was fabricated by DC PACVD (plasma-assisted chemical vapor deposition) on a silicon substrate. The thickness of the diamond film was about 35 μm. The silicon substrate was etched out with fluoric acid to measure the thermal diffusivity of the diamond film. Because the diamond film is optically transparent, DGF (dry graphite film) spray was used to blacken the surfaces. The effect of the graphite film was ignored. To sense the temperature variation, an E-type thermocouple (chromel–constantan; diameter, 0.002 in.) was attached by spot welding on the metal specimen and silver paste on the nonmetal specimen. The size of each specimen was approximately $5 \times 10\ mm^2$. The specimen was enclosed in a copper cylinder (diameter, 25 mm) for radiation shielding. The variation of temperature was recorded and analyzed by personal computer.

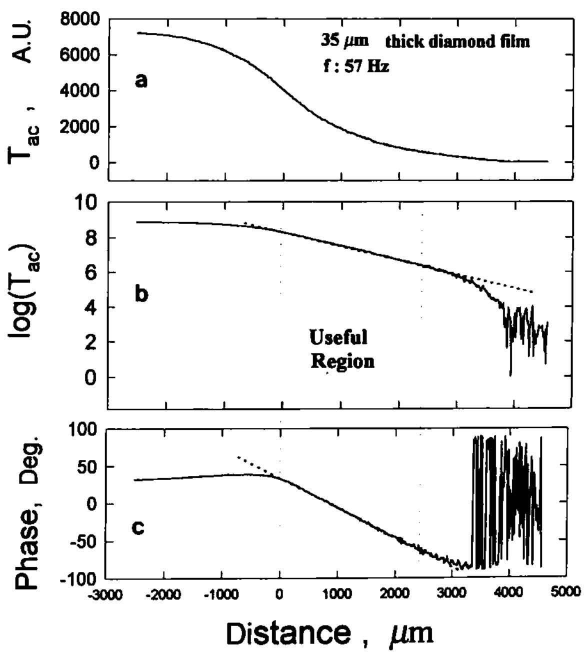

Fig. 2. Typical plots of the (a) amplitude of the AC temperature wave, (b) logarithm of the amplitude, and (c) phase of the AC temperature wave as a function of the distance from the thermocouple to the edge of the mask. To determine the thermal diffusivity, the slopes of the dashed line in b and c are used.

4. RESULTS AND DISCUSSION

Raw data from the diamond film are shown in Fig. 2, as an example. According to Eq. (7), for $1/\omega\tau_e \ll 1$, the thermal diffusivity of the diamond film can be determined from Fig. 2b the slope of $\ln(|T_{ac}|)$ vs position because the slope, $k_r = \sqrt{\pi f/\alpha_r}$. However, even if $\alpha/\pi l^2 < f \ll \alpha/\pi d^2$, the thermal diffusivity calculated from these slopes tends to decrease for increasing chopping frequency. Moreover, the thermal diffusivity determined from the phase component is always larger than the one determined from the amplitude component and tends to increase with increasing frequency. This means that frequency effects require the use of results from both components. To estimate the frequency effects we deduced the thermal diffusivity through vectorial calculation of two results.

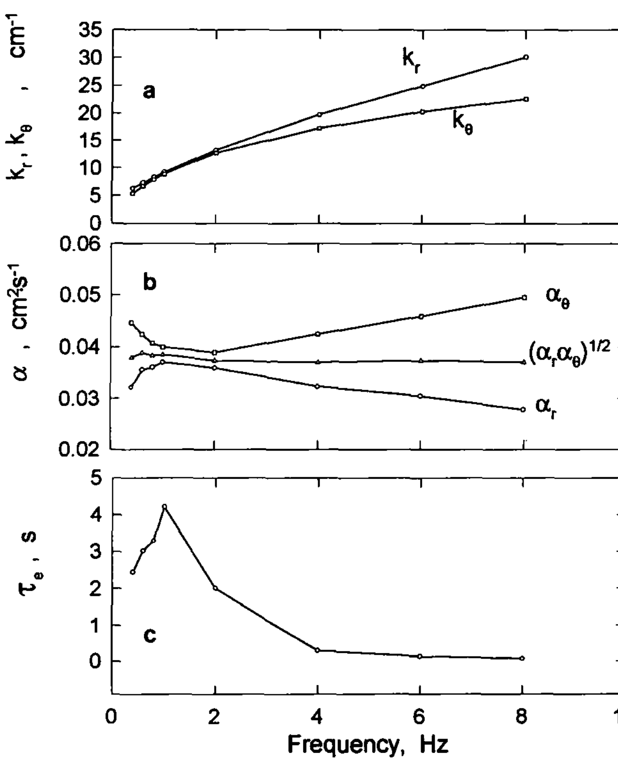

Fig. 3. Results for 50-μm-thick SUS-304 foil: (a) variation of thermal wave numbers k_r and k_θ with chopping frequency; (b) thermal diffusivities calculated from k_r and k_θ; (c) apparent external relaxation time for the SUS-304 foil obtained using Eq. (14).

The measured results obtained for the SUS-304 foil as a function of chopping frequency are plotted in Fig. 3. The variation of the apparent diffusivity values at lower frequencies in Fig. 3b is due to the longer thermal wavelength ($\lambda_{thermal} = 2\pi/k$) than sample length l. In this case, the wave scheme solution of Eq. (4) is not applicable. However, the deduced result $\sqrt{\alpha_r \alpha_\theta}$ in Fig. 3b is still independent of frequency variation. Figure 3c is the external thermal relaxation time constant (τ_e) for frequency variation as defined in Eq. (5). From this we can determine the range of proper chopping frequency for measuring the thermal diffusivity of the sample. Since the peak around 1 Hz in Fig. 3c represents the lower limit of the frequency satisfying $\lambda_{thermal} \leqslant l$, the acceptable frequency range for the SUS-304 foil is about $f > 1$ Hz. From these two α's in the range of $f > 1$ Hz, we can determine the apparent thermal diffusivity of 0.0371 $cm^2 \cdot s^{-1}$ with a deviation of $\pm 3\%$.

This method was tested on other foil-shaped samples. The deduced result of two diffusivities, α_r and α_θ, is relatively independent of frequency variation also. From these results we could confirm that, to measure the thermal diffusivity of foil-shaped materials with the AC calorimetric method, it is necessary to measure the amplitude and phase of the AC thermal wave rather than measure the amplitude component only.

5. SUMMARY

Thermal diffusivities of foil-shaped materials were measured using the AC calorimetric method. To determine the thermal diffusivity of a specimen, vectorial analysis of the amplitude and phase of the AC temperature signal was used. The results of this method are more reliable than those of one-component (amplitude or phase of the AC temperature signal) methods and relatively independent of frequency variation. Therefore, the vectorial analytic method for analyzing photoirradiation-type AC calorimetric experiments is a useful technique for measuring thermal diffusivities of foil-shaped materials.

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