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Analysis of Two-Dimensional Effect in the Measurement of Thermal Diffusivity of Thin Films with an AC Calorimetric Method[1]

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An ac calorimetric method for measuring the thermal diffusivity of thin-film materials has been widely applied. In the application of this method, the systematic errors caused by the heat loss effect, the edge reflection effect, etc., have been analyzed and corresponding correction methods have been developed. But when measuring films with low thermal diffusivity or with thickness comparable to the thermal diffusion length, a two-dimensional effect which will also result in a systematic error of the measurement is present. In this paper, the mechanism of two-dimensional heat conduction within a thin sample which is supplied a periodic heat flux by a chopped light beam is analyzed. A numerical analysis method is developed to study the effect of the two-dimensional heat conduction on the measured thermal diffusivity values. The relations between the measured thermal diffusivity and independent parameters such as frequency, thickness of sample, width of light spot, etc., are demonstrated to indicate the two-dimensional effect. The experimental precondition for minimizing the systematic error caused by the two-dimensional effect is determined. In addition, the analysis method presented in this paper should be useful for more difficult problems such as error estimation of the thermal diffusivity measurement of coatings or composite films.

KEY WORDS: ac calorimetric method; thermal diffusivity; thin film; two-dimensional effect.

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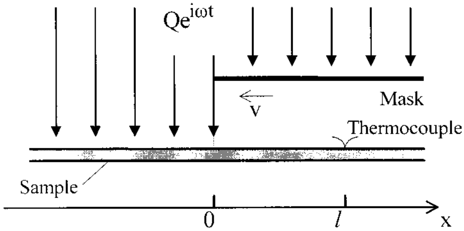

Fig. 1. Principle of the ac calorimetric method.

1. INTRODUCTION

The principle of the ac calorimetric method for thermal diffusivity measurements in thin films is shown in Fig. 1 [1]. A modulated uniform light beam is applied perpendicularly to the surface of the platelike thin-film sample that is partly shadowed by a mask moving along the $-x$ direction. The light energy absorbed by the surface of the sample is converted into a uniform heat flux $Qe^{i\omega t}$, and produces a temperature wave that propagates along the sample length. A fine thermocouple with a diameter smaller than the thermal diffusion length is attached to a point of the sample surface lying under the mask. The ac temperature amplitude $|T(l, t)|$ and phase $\arg[T(l, t)]$ are measured as a function of the distance l. The thermal diffusivity α along the sample length can be obtained as follows [1]:

Amplitude method:

$$\alpha = \frac{1}{2}\omega \left[\frac{d \ln |T(l, t)|}{dl}\right]^{-2} \tag{1}$$

Phase method:

$$\alpha = \frac{1}{2}\omega \left\{\frac{d \arg[T(l, t)]}{dl}\right\}^{-2} \tag{2}$$

When studying the two-dimensional effect on the measurement, a two-dimensional heat conduction model should be considered.

2. BASIC THEORY

Under the above heating condition, the temperature wave in the sample is

$$T = A_d(\vec{r})\, e^{i[\omega t + \varphi(\vec{r})]} \tag{3}$$

The heat conduction equation is

$$\nabla^2 T = \frac{1}{\alpha}\frac{\partial T}{\partial t} \tag{4}$$

By Eqs. (3) and (4), we get [2]

$$\begin{cases} \nabla^2 A_d - A_d(\nabla\varphi)^2 = 0 \\ \nabla\cdot(A_d\nabla\varphi) + \nabla A_d\nabla\varphi - \dfrac{\omega}{\alpha}A_d = 0 \end{cases} \tag{5}$$

The first-type boundary condition is

$$T_b = A_b e^{i(\omega t + \varphi_b)} \Rightarrow \begin{cases} A_d = A_b \\ \varphi = \varphi_b \end{cases} \tag{6}$$

The second-type boundary condition is

$$-\lambda\frac{\partial T}{\partial n} = q_b e^{i(\omega t + \varphi_b)} \Rightarrow \begin{cases} \left|-\lambda\dfrac{\partial A_d}{\partial n}\right| = q_b\,|\cos(\varphi - \varphi_b)| \\ \left|-\lambda A_d\dfrac{\partial\varphi}{\partial n}\right| = q_b\,|\sin(\varphi - \varphi_b)| \end{cases} \tag{7}$$

And the third-type boundary condition is

$$-\lambda\frac{\partial T}{\partial n} = hT \Rightarrow \begin{cases} -\lambda\dfrac{\partial A_d}{\partial n} = hA_d \\ -\lambda A_d\dfrac{\partial\varphi}{\partial n} = 0 \end{cases} \tag{8}$$

where q_b is the absorbed heat flux amplitude and φ_b is the heat flux phase.

3. NUMERICAL MODEL

In order to analyze the effect of two-dimensional heat conduction on the measurement of the thermal diffusivity of thin films, we use the numerical model shown in Fig. 2. In the figure, the y direction is normal to the surface of the sample and the x direction is parallel to the length of the sample. The sample length is sufficiently long to ensure that there is no edge reflection of the temperature wave at the two ends.

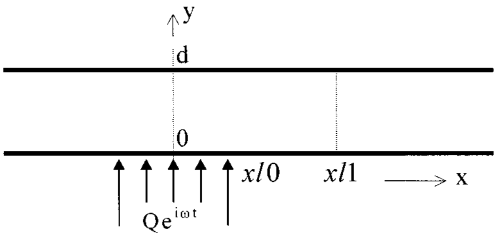

Fig. 2. Analytical model of the two-dimensional effect.

Some parameters are defined as follows:

$$L_0 = \sqrt{\frac{2\alpha}{\omega}}, \qquad A_0 = \frac{2q_b}{\omega c d}, \qquad He = \frac{h}{\sqrt{\omega c \lambda / 2}} \tag{9}$$

where L_0 is the thermal diffusion length, A_0 is the characteristic temperature, and He is the dimensionless heat transfer coefficient.

The corresponding dimensionless amplitude and phase equations of Eq. (5) are

$$\begin{cases} \dfrac{\partial^2 A}{\partial X^2} + \dfrac{\partial^2 A}{\partial Y^2} - A\left[\left(\dfrac{\partial \varphi}{\partial X}\right)^2 + \left(\dfrac{\partial \varphi}{\partial Y}\right)^2\right] = 0 \\ \dfrac{\partial}{\partial X}\left(A\dfrac{\partial \varphi}{\partial X}\right) + \dfrac{\partial}{\partial Y}\left(A\dfrac{\partial \varphi}{\partial Y}\right) + \dfrac{\partial A}{\partial X}\dfrac{\partial \varphi}{\partial Y} + \dfrac{\partial A}{\partial X}\dfrac{\partial \varphi}{\partial Y} - 2A = 0 \end{cases} \tag{10}$$

By Eqs. (7) and (8), the boundary conditions can be defined as

$$\begin{cases} -\dfrac{\partial A}{\partial Y} = YL\,|\cos\varphi|, \\ -A\dfrac{\partial \varphi}{\partial Y} = YL\,|\sin\varphi|, \end{cases} \qquad Y = 0, \quad 0 \leqslant X \leqslant XL0 \tag{11}$$

$$\begin{cases} \dfrac{\partial A}{\partial Y} = -HeA, \\ \dfrac{\partial \varphi}{\partial Y} = 0, \end{cases} \quad Y = 0 \qquad \text{and} \qquad \begin{cases} \dfrac{\partial A}{\partial Y} = HeA, \\ \dfrac{\partial \varphi}{\partial Y} = 0, \end{cases} \quad Y = YL \tag{12}$$

where $YL = d/L_0$, $XL0 = xl0/L_0$, $A = A_d/A_0$, $X = x/L_0$, $Y = y/L_0$.

4. ANALYSIS OF CALCULATION RESULTS

4.1. Measurement with No Heat Loss

According to Eqs. (10) to (12), the independent parameters in the measurement of thermal diffusivity are *YL*, *XL*0, and *He*. As an example, when $He = 0$, $YL = 0.5$ and $XL0 = 0.2$, and the temperature distribution on the surface of the sample is calculated. Installing the thermocouple on both the top and bottom surfaces is considered in the calculations. The α^*/α versus x curves deduced from the temperature distribution are plotted in Fig. 3, where α^* is the calculated thermal diffusivity with the two-dimensional effect. In an experiment, it will be the actually measured thermal diffusivity.

Figure 3 gives the calculated curves of α^*/α versus x based on the amplitude method with no heat loss. The curves calculated by the phase method are almost the same as those shown in this figure. As with many other caculations, this shows that when there is no heat loss, the two-dimensional effect is caused by the asymmetrical heating of the sample by the light beam. If the thermocouple is installed far enough from the light beam boundary, the effect will disappear.

According to the above analysis, the point $xl1$ in Fig. 2 is defined to meet the following condition: if the thermocouple is placed at x and $x > xl1$, then the relative systematic error $|(\alpha^* - \alpha)/\alpha|$ caused by the two-dimensional effect will be less than 2%. Since neglect of the heat loss means $He = 0$, $xl1$ is only determined by the independent parameters *YL* and

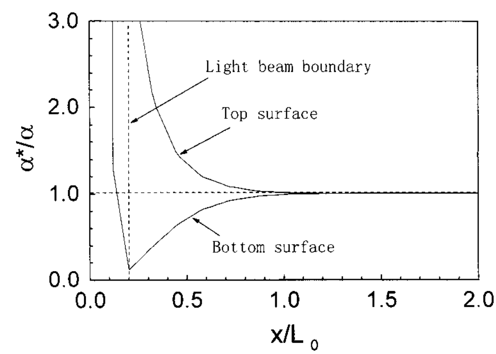

Fig. 3. Calculated curves of α^*/α versus x by the amplitude method with no heat loss.

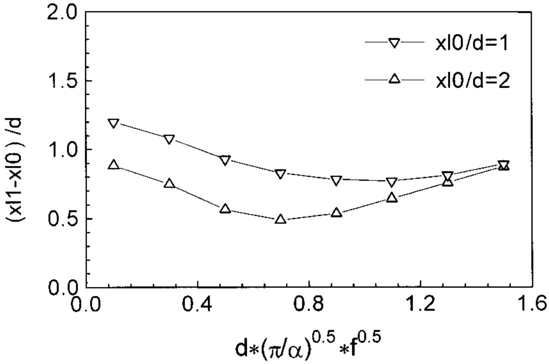

Fig. 4. Curves of Eq. (13).

$XL0$. The relationship between $xl1$, YL, and $XL0$ can be expressed in a functional form:

$$\frac{xl1 - xl0}{d} = f\left(\frac{xl0}{d}, YL\right) \tag{13}$$

where f relates to Eqs. (10) to (12). The calculated curves are shown in Fig. 4.

Figure 4 shows that $xl1 - xl0$ is determined by YL and $XL0$. As the value of YL becomes smaller, the value of $xl1 - xl0$ will first decreases, then increase. Thus, measurments at low frequency will not eliminate the two-dimensional effect caused by the asymmetrical heating of the sample. The figure also shows that if $2xl0 > 4d$, then $xl1 - xl0 < d$. This means that in a measurement, the thermocouple should not be placed too close to the boundary of the light beam. If the thermocouple is placed outside the range $xl1 - xl0$, the two-dimensional effect caused by the asymmetrical heating of the sample can be neglected.

4.2. Measurement with Heat Loss

Under the conditions $He = 0.5$, $YL = 0.5$, and $XL0 = 0.2$, the α^*/α versus x curves are calculated and plotted in Fig. 5 as an example.

The systematic error caused by heat loss can be corrected by the following equations with the assumption of one-dimensional heat conduction [3]:

Amplitude method:

$$\frac{\alpha}{\alpha^*_{\text{Amp}}} = \frac{1}{\omega\tau_e} + \sqrt{1 + \left(\frac{1}{\omega\tau_e}\right)^2} \tag{14}$$

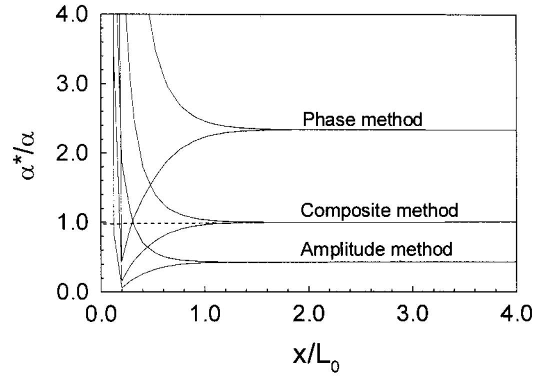

Fig. 5. Calculated curves of α^*/α versus x with heat loss.

Phase method:

$$\frac{\alpha}{\alpha^*_{\mathrm{Phr}}} = \frac{1}{\omega\tau_e} + \sqrt{1 + \left(\frac{1}{\omega\tau_e}\right)^2} \tag{15}$$

Composite method:

$$\alpha = \sqrt{\alpha^*_{\mathrm{Amp}}\alpha^*_{\mathrm{Phr}}} \tag{16}$$

Here α^*_{Amp} is the measured thermal diffusivity by the amplitude method, α^*_{Phr} is the measured thermal diffusivity by the phase method, and τ_e is the heat loss relaxation time defined as

$$\tau_e = \frac{cd}{h} \tag{17}$$

Considering the definition of the parameters He and τ_e, we obtain

$$\frac{1}{\omega\tau_e} = \frac{He}{YL} \tag{18}$$

If $He = 0.5$ and $YL = 0.5$, then by Eqs. (14) to (16), we get $\alpha^*_{\mathrm{Amp}}/\alpha = 0.414$, $\alpha^*_{\mathrm{Phr}}/\alpha = 2.414$, and $\alpha^*/\alpha = 1$. By a two-dimensional calculation, when $x/L_0 > xl1/L_0$, we get $\alpha_{\mathrm{Amp}}/\alpha = 0.43$, $\alpha_{\mathrm{Phr}}/\alpha = 2.34$, and $\alpha^*/\alpha = 1$. It is obvious that the results are different. This means when there is heat loss, the two-dimensional effect cannot be neglected even if the thermocouple is located far from the light beam boundary.

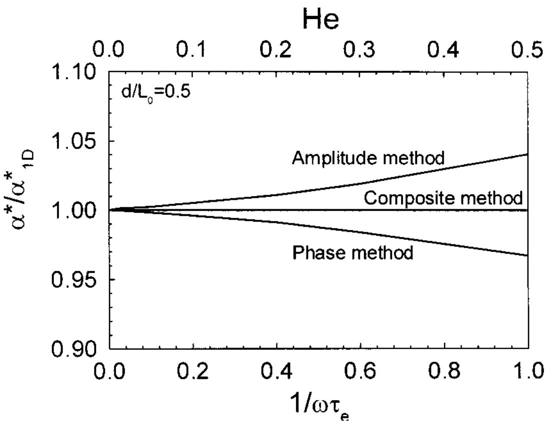

Fig. 6. α^*/α^*_{1D} versus $1/\omega\tau_e$.

Figures 6 and 7 show the two-dimensional effect caused by heat loss, where α^* is the calculated thermal diffusivity for $x/L_0 > xl1/L_0$, with no two-dimensional effect caused by asymmetrical heating. Here, α^*_{1D} is obtained from Eqs. (14) to (16), which are deduced from the one-dimensional heat conduction model.

Figure 6 shows that heat loss intensity (the value of He or $1/\omega\tau_e$) will influence the two-dimensional effect. If $He = 0$ and $x/L_0 > xl1/L_0$, there is no two-dimensional effect. But when $He \neq 0$, the two-dimensional effect exists over the whole length of the sample. The value measured by the amplitude method will be higher than the actual value, and the value measured by the phase method will be lower than the actual value. The composite method can correct not only the systematic error caused by heat

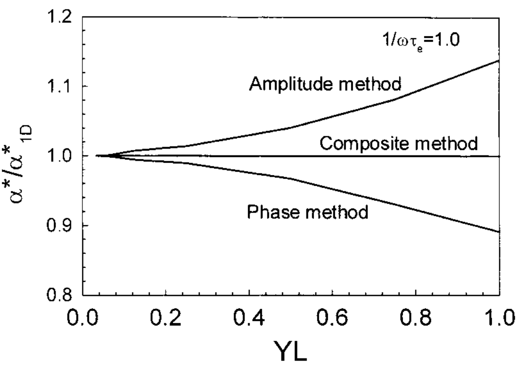

Fig. 7. α^*/α^*_{1D} versus YL.

loss, but also the error caused by the two-dimensional effect induced by heat loss.

Figure 7 shows that as the sample dimensionless thickness YL increases, the two-dimensional effect induced by heat loss becomes more and more evident.

4.3. Elimination, Minimization, or Correction of the Two-Dimensional Effect

From the above calculations and analysis, some useful methods of eliminating, minimizing, or correcting the two-dimensional effect can be obtained.

Installing the thermocouple at $x/L_0 > xl1/L_0$ will avoid the two-dimensional effect induced by asymmetrical heating. If $2xl0 > 4d$, then the condition $xl1 - xl0 < d$ should be satisfied.

We should try to minimize the heat loss as much as possible. If $He < 0.1$ and $YL < 0.5$, the two-dimensional effect induced by heat loss can be neglected.

If the heat loss intensity is large, the composite method should be used in processing the experiment data.

5. CONCLUSION

In the measurement of the thermal diffusivity of a thin film, the two-dimensional effect exists because the modulated light beam heats only one side of the sample and because there is heat loss. The two-dimensional effect due to the first cause exists only in some areas of the sample, and can be neglected if the thermocouple is installed far enough from the light beam boundary. The two-dimensional effect due to the second cause can be corrected by the composite method.

NOMENCLATURE

c	specific heat per volume ($J \cdot m^{-3} \cdot K^{-1}$)
d	sample thickness (m)
h	effective heat transfer coefficient ($W \cdot m^{-2} \cdot K^{-1}$)
λ	thermal conductivity ($W \cdot m^{-1} \cdot K^{-1}$)
ω	angular frequency
n	surface normal
$xl0$	half-width of the light beam (m)
$xl1$	minimum distance from the center of the light beam in which two-dimensional effect must be considered in measurements (m)

A_d	dimensional temperature wave amplitude (K)
A_b	amplitude of the boundary temperature
φ	temperature wave phase
φ_b	phase of the boundary temperature
α	actual thermal diffusivity ($m^2 \cdot s^{-1}$)
α^*	measured thermal diffusivity with the two-dimensional effect ($m^2 \cdot s^{-1}$)
T	temperature/temperature wave (K)
T_b	boundary temperature
L_0	thermal diffusion length
A_0	characteristic temperature
He	dimensionless heat transfer coefficient
$XL0$	dimensionless half-width of the light beam
YL	dimensionless sample thickness

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