International Journal of Thermophysics, Vol. 21, No. 2, 2000

Correction of Edge Effect in AC Calorimetric Method for Measuring Thermal Diffusivity of CVD Diamond Films[1]

Y. Gu,[2,5] F. Liao,[3] and I. Hatta[4]

In the thermal diffusivity measurement of a CVD diamond film using an ac calorimetric method, the reflection of an ac temperature wave at the edge of the film sample should be considered due to the limited length of the sample and its high thermal diffusivity, i.e., the edge effect. In this case, the measured thermal diffusivity is given as a function of frequency. The relation between the measured thermal diffusivity and the frequency is represented as an analytical expression. The real thermal diffusivity is obtained by correcting the edge effect by two means. One is an iterative method using the directly measured edge length of the sample to fit the analytical expression. The other is a parameter estimation method by which a simplex method is used to estimate the edge length and the real thermal diffusivity. Thermal diffusivities of two diamond films were measured, and data were analyzed using the above methods. The result shows that the parameter estimation method is relatively accurate and convenient in processing test data.

KEY WORDS: ac calorimetric method; CVD diamond film; edge effect; parameter estimation; thermal diffusivity.

1. INTRODUCTION

An ac calorimetric method for thermal diffusivity measurement of thin films has been developed successfully in recent years [1]. A chopped light beam

---

[1] Paper presented at the Fifth Asian Thermophysical Properties Conference, August 30–September 2, 1998, Seoul, Korea.

[2] Department of Engineering Mechanics, Tsinghua University, Beijing 100084, People's Republic of China.

[3] Beijing Institute of Spacecraft System Engineering, P.O. Box 9628, Beijing 100086, People's Republic of China.

[4] Department of Applied Physics, Nagoya University, Nagoya 464-01, Japan.

[5] To whom correspondence should be addressed.

0195-928X/00/0300-0487$18.00/0

is applied to the surface of the platelike sample of thin-film partly shadowed by a mask moved along the surface. A modulated uniform light beam irradiated on the surface of the sample converts a uniform heat flux $Qe^{i\omega t}$. A temperature wave with the same frequency as the heat flow propagates along the surface of the sample. A fine thermocouple with a diameter smaller than the thermal diffusion length is attached to a part of the sample lying under the mask. The ac temperature amplitude is measured as a function of the distance between the thermocouple and the center of the heat source. By measuring the relation of the amplitude with the distance, the thermal diffusivity in the direction of propagation can be given by

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

where $|T(x,t)|$ is the amplitude of the temperature wave and ω is the angular frequency of the heat flux.

For the case where the sample has a high thermal diffusivity and limited length—for example, a diamond film—usually the temperature wave will reflect at the ends of the sample. This reflection will cause the measured thermal diffusivity from Eq. (1) to vary from the real thermal diffusivity. This is defined as an edge effect. In order to obtain the real thermal diffusivity, a correction for the edge effect should be carried out.

In this paper, two methods of correction of the edge effect are presented. The first is an iterative method using the directly measured edge length of the sample to fit the analytical expression which gives the relation between the real thermal diffusivity and the measured value with the edge effect. The second is a parameter estimation method developed recently. The second method is recommended because of its accuracy and convenience. The results of experiments on CVD diamond films are reported.

2. PRINCIPLE

As shown in Fig. 1, an ac thermal energy with uniform strength $Qe^{i\omega t}$ was applied by chopper light irradiation.

The distance between the center of the irradiated region and the thermocouple is denoted by l. For the case where the sample has a high thermal diffusivity and limited length, when the temperature wave reflects at the edge denoted by $-n$, the edge effect should be taken into account. The real thermal diffusivity of the sample is different from the apparent value and can be determined by the following equations [2]:

$$\frac{\alpha}{\alpha_A} = \left\{1 - \frac{2e^{-2k(n-l)}[\cos 2k(n-l) + \sin 2k(n-l) + e^{-2k(n-l)}]}{1 + 2e^{-2k(n-l)}\cos 2k(n-l) + e^{-4k(n-l)}}\right\}^2 \tag{2}$$

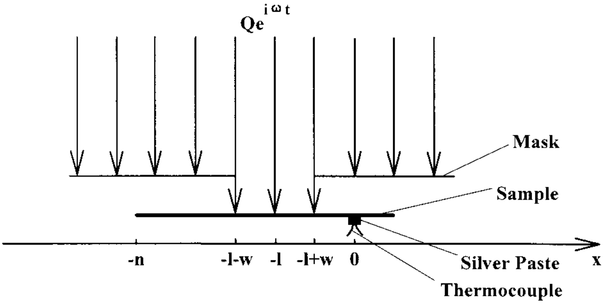

Fig. 1. Schematic view of ac calorimetric method.

where $k=\sqrt{\omega/(2\alpha)}$ is the decay constant of the ac temperature wave, α is the real thermal diffusivity, and α_A is the apparent thermal diffusivity obtained by Eq. (1) in the experiment.

There are two ways to correct the edge effect and calculate α with Eq. (2):

1. After measuring the value of $(n-l)$ directly in the experiment, obtain α by an iterative calculation with data on a series of apparent thermal diffusivities and frequencies f, i.e., ω [3].
2. Apply the parameter estimation method to estimate $(n-l)$ and α simultaneously with data from a series of apparent thermal diffusivities α_A and frequencies f, i.e., ω.

For the parameter estimation method [4, 5], we define the object function J as

$$J=\sum_{i=1}^{N}\left[\alpha_{Ai}-f(n-l,\alpha,\omega_i)\right]^2 \tag{3}$$

and

$$f(n-l,\alpha,\omega)=\alpha\left\{1-\frac{2e^{-2k(n-l)}\left[\cos 2k(n-l)+\sin 2k(n-l)+e^{-2k(n-l)}\right]}{1+2e^{-2k(n-l)}\cos 2k(n-l)+e^{-4k(n-l)}}\right\}^{-2} \tag{4}$$

where $i=1, 2, \ldots, N$, and N is the number of measurements.

Using Eq. (3), the values of $n-l$ and α for which the variable J obtains a value less than a previously set maximum can be found by using parameter estimation programs. These values are the desired optimal values of $n-l$ and α.

3. EXPERIMENT AND RESULTS

The ac light was supplied by an optical system with a halogen lamp as a light source. The sample was placed in a vacuum chamber with a thermocouple attached on the surface to detect the temperature signal. The test frequency f ($\omega = 2\pi f$) ranged from 0.4 to 8.0 Hz. The heat loss was considered to be negligible.

Free-standing CVD diamond films were tested. The thermal diffusivities of two samples, A and B, were measured. The SEM images of these two samples are shown in Figs. 2 and 3. The amplification coefficients of the two images are both 600:1.

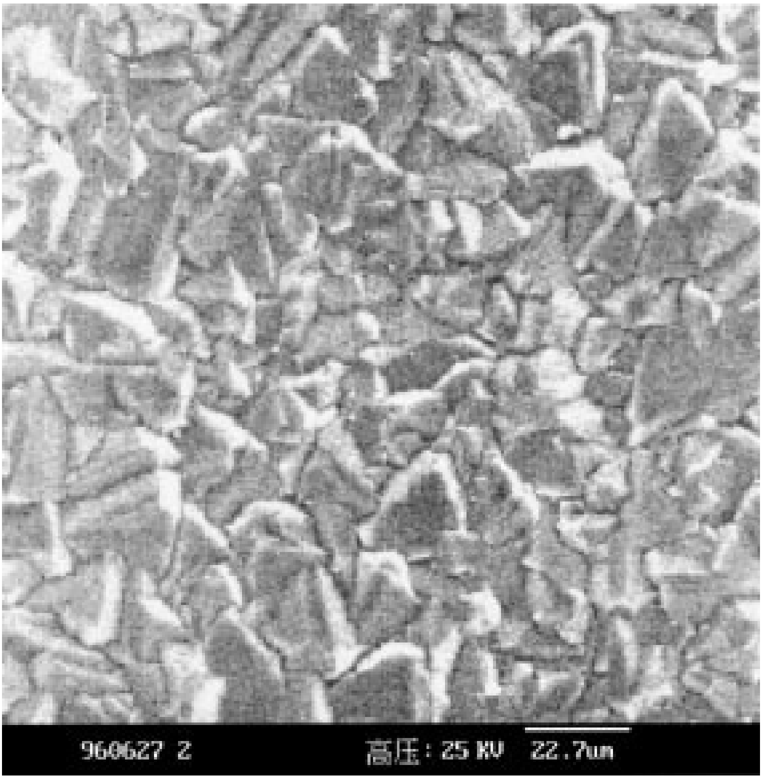

Fig. 2. SEM image of sample A.

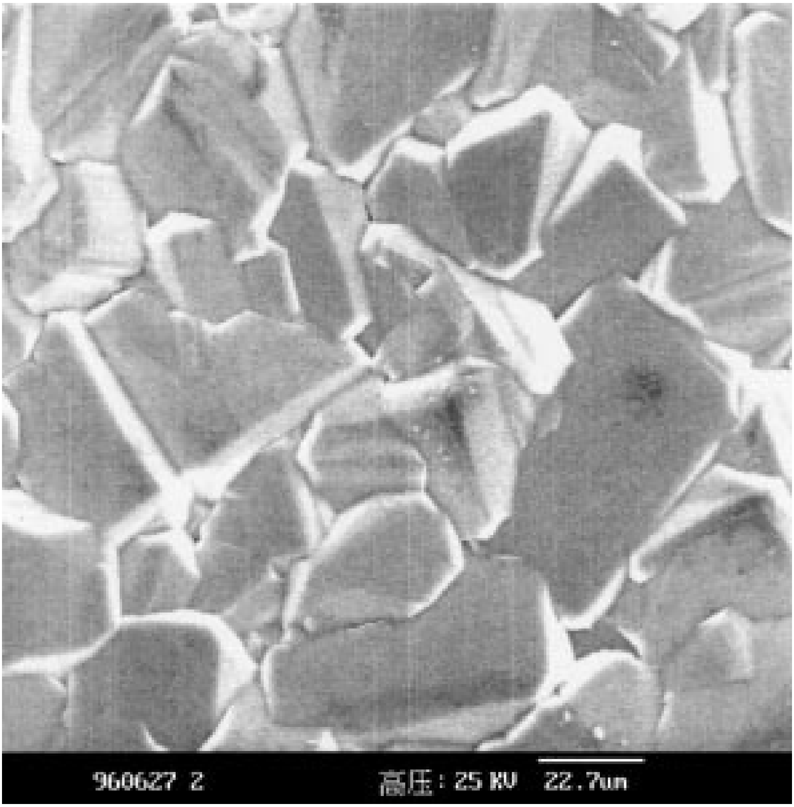

Fig. 3. SEM image of sample B.

Table I presents the measured and calculated results for samples A and B. The thermal diffusivity of B is much larger than that of A. This result is expected since the grain size of film B is much larger than A as shown in Figs. 2 and 3.

Table I. Measured and Calculated Results for Diamond Films

Sample	Thickness (μm)	Method 1 (measured)		Method 2 (estimated)	
		$n-l$ (mm)	α ($cm^2 \cdot s^{-1}$)	$n-l$ (mm)	α ($cm^2 \cdot s^{-1}$)
A	50	11.80	2.99	12.89	2.98
B	140	10.44	6.12	11.96	6.30

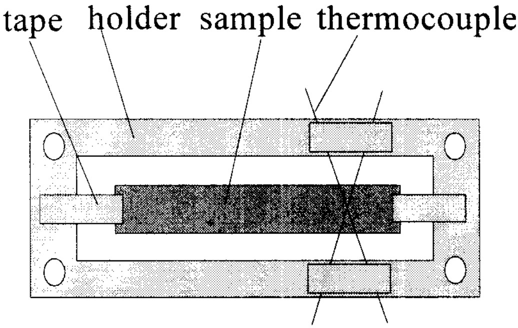

Fig. 4. Installation of sample on holder.

From Table I it is noted that the estimated $n-l$ is slightly greater than the measured value. After reviewing the method of installing the sample on the holder in the experiment, tapes were attached to the sample to suspend it on the holder as shown in Fig. 4.

Obviously the tapes reduce the edge effect, since, when the temperature wave propagates at the sample end, part of the wave can disseminate into the tape and not be reflected. The effect is analogous to prolonging the sample somewhat. The estimated value of $n-l$ includes this installation effect. In Fig. 5 the results of method 1 (measuring $n-l$ directly) and method 2 (parameter estimation) are compared.

The data points (stars) from method 2 fit the theoretical curve calculated from Eq. (2) better than those from method 1. Thus, the use of the parameter estimation method to determine the thermal diffusivity α is

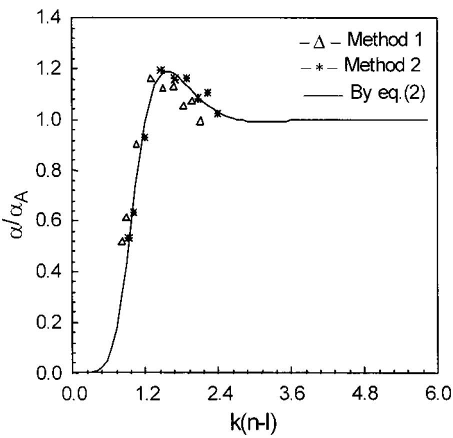

Fig. 5. Edge reflection effect.

more accurate than directly measuring $n - l$ and then iterating to find the value α.

4. CONCLUSIONS

1. An ac calorimetric method can be successfully used to measure the thermal diffusivity of CVD diamond films. Because of the finite length of the sample and its high thermal diffusivity, an edge effect correction to the measured value should be made.

2. Two ways of correcting the edge effect are presented. It is shown that the parameter estimation method is more accurate and convenient than the iterative method with direct measurements of the value of $n - l$.

ACKNOWLEDGMENTS

The authors thank Prof. Li Hou and Erkai Liu of the Institute of Synthetic Crystals for supplying the samples of CVD diamond films. This work has been supported by the National Natural Science Foundation of China and the Japan Society for the Promotion of Science.

REFERENCES

1. I. Hatta, Y. Sasuga, R. Kato, and A. Maesono, *Rev. Sci. Instrum.* **56**:1643 (1985).
2. Yuqin Gu and I. Hatta, *Jpn. J. Appl. Phys.* **30**:1137 (1991).
3. Yuqin Gu and Lixin Yu, *Diamond Films Technol.* **6**:23 (1996).
4. Yuqin Gu, *J. Eng. Thermophys.* **8**:83 (1987) [in Chinese].
5. J. V. Beck and K. J. Arnold, *Parameter Estimation in Engineering and Science* (Wiley, New York, 1976).