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THERMAL DIFFUSIVITY MEASUREMENT OF THIN FILM BY AC CALORIMETRIC TECHNIQUE

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ABSTRACT

A new method to measure thermal diffusivity of a thin sample in a direction parallel to the plane has been developed using light-irradiated ac calorimetric technique. By this method thermal diffusivity of stainless steel, nickel, alumina and silicon in 0.1 to 0.3 mm thickness range are measured and these results are in good agreement with the data listed in TPRC data book. Thermal diffusivity of some special materials such as anisotropic organic semiconductor and amorphous metal ribon is obtained also. The measuring conditions are as follows; sample size is 9x4 mm^2 in area and 0.05 to 0.3 mm in thickness, temperature range examined is from room temperature to 500 °C, chopping frequency of light is 1.0 to 16 Hz.

INTRODUCTION

Recently electronic industry needs the data of thermal diffusivity for a very thin plate or film, so that traditional methods can not be applied for such thin plates.

Usually traditional steady state methods require a sample of more than 5 mm thickness. Laser flash method; which is applicable to relatively thin sample, requires sample of at thinnest 0.5 mm thickness.

We developed a method using ac calorimetric technique to measure thermal diffusivity in a direction parallel to a plane of thin samples. This method is not basically limited in the low bound of the thickness of sample and rather has higher sensitivity as sample is thinner.

MEASURING METHODS

A schematic diagram of our measuring system is shown in Figs.1 and 2. A thin wire thermocouple(6) is spot-welded or glued with silver-paste on the sample(1) to measure ac temperature response (T_{ac}). A sample with a mask plate(2) which can be moved by a micrometer(5) is irradiated by light of halogen lamp(4). Ac temperature is amplified by lock-in amplifier(8). Reference signal of lock-in amplifier is provided by photo-detector(7).

The relation of the ac temperature ($\dot{T}_{ac}$) and the displacement of the mask (L) can be written in the following equation 1:[1]

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$$ac = \frac{QR}{\cosh[kL(1+i)]+RSk(1+i)\sinh[kL(1+i)]} , \qquad (1)$$

where S is thermal conductivity along the plane, Q is heat energy supplied by light irradiation, R is thermal resistance between a sample and thermal bath. And k is the reciprocal of the thermal diffusion length:

$$k = \sqrt{\pi f / D} , \qquad (2)$$

where f is measuring frequency and D is thermal diffusivity.

Equation 1 can be rewritten as follows when R and f are large($kL \gtrsim 1$, $RSk > 1$):

$$T_{ac} = \frac{\sqrt{2}\,Q}{S\,k}\, e^{-kL-i(kL+\pi/4)} . \qquad (3)$$

Absolute value of T_{ac} is given by Eqs.4 and 5,

$$T_{ac} = \frac{\sqrt{2}\,Q}{S\,k}\, e^{-kL} . \qquad (4)$$

Therefore,

$$\ln|T_{ac}| = \ln\frac{\sqrt{2}\,Q}{S\,k} - kL . \qquad (5)$$

Equation 5 indicates that $\ln|T_{ac}|$ is proportional to L and k is given as the slope of $\ln|T_{ac}|$ versus L plot, which is shown in Fig.3 for alumina. Caluculating the

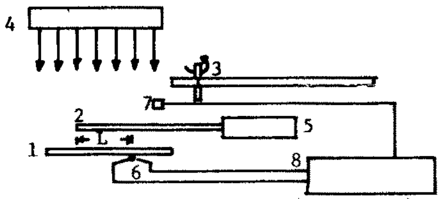

Fig.1. Schematic diagram (side view). See figure caption of Fig.2.

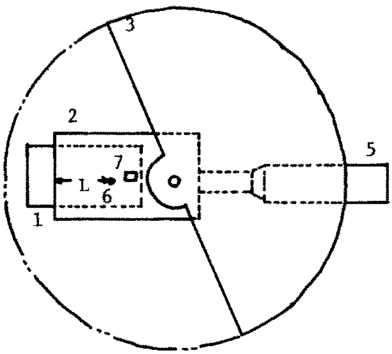

Fig.2. Schematic diagram (top view). Sample-1;mask-2;chopper-3;light source-4;micrometer-5;thermocouple-6; photo-detector-7;lock-in amplifier-8.

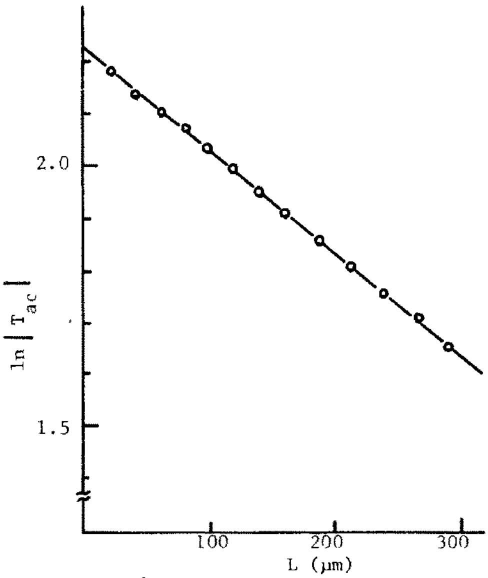

Fig.3. $\ln|T_{ac}|$ versus L plot. Sample;alumina,thickness:0.1mm,chopping frequency;10 Hz.

value from the slope of the straight line and substituting k value into Eqs.2, hermal diffusivity of an unknown sample can be obtained.

RESULTS AND DISCUSSION

The results of the thermal diffusivity measurement of stainless steel, nickel, alumina and silicon at room temperature are tabulated in table 1. They are in good agreement with laser flash method and also TPRC[2] data within ±5%. Thermal diffusivity of organic semiconductor which has anisotropy in its plane was measured. Thermal diffusivity of X direction in the plane is 0.014 cm^2/sec and in tne Y direction perpendicular to X is 0.0058 cm^2/sec. The difference of the thermal diffusivity between X and Y direction seems to be due to its molecular orientation. The present method can be applied for a thin sample to the measurement of anisotropy in thermophysical properties, but laser flash method not.

Fig.4 and table 2 show for amorphous metal ribon. In first run a sample was heated from room temperature to 192 °C below its recrystallization temperature. After cooling from 192°C, in second run up to 468 °C over the recrystallization temperature the thermal diffusivity of the sample was measured. The results of the first and second runs agree in the each other precisely. The recrystallization temperature which was obtained from heat capacity anomaly by ac calorimetric method is 459.5 °C. After recrystallization, it has a much different value from the other sample.

Fig.5 and table 3 show thermal diffusivity of nickel under the heating run from room temperature to about 500 °C. The present data of first run and second run are in good agreement within ±5%. But data is different from TPRC data only at room temperature. This difference seems to be due to sample quality.

SAMPLE	THICKNESS (mm)	MEASURED RESULT, D(cm^2/sec) FREQUENCY, f(Hz) f_1	f_2	f_3	LITERATURE LSAER FLASH METHOD	TPRC
stainless steel	0.05	4 Hz 0.033	9 Hz 0.033	16 Hz 0.033	0.033	0.032-0.037
nickel	0.20		9 Hz 0.200		0.200	0.15-0.25
alumina	0.10	9 Hz 0.075	12 Hz 0.075	16 Hz 0.073	0.078	0.068-0.090
silicon	0.30	1 Hz 0.845				0.820-0.950
organic semiconductor	0.05	1 Hz 0.014	4 Hz 0.013			
	0.05	1 Hz 0.0058				

Table 1. Thermal diffusivity results of various samples.

CONCLUTIONS

The feature of developed instrument is as follows;

1. Be able to measure in a very thin sample.
2. Be able to check anisotopy along plane.
3. Be able to measure even in a translucent sample such as alumina.

Study for multi layer samples is now in progress.

ACKNOWLEDGMENT

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REFERENCES

1. I.Hatta and A.J. Ikushima, Jpn.J. Appl.Phys. 20(1981)
2. Thermophysical properties of matter. The TPRC Data series Volume 10.

Temperature(°C)		20	97	192	288	386	468
Thermal diffusivity	1st	0.025	0.028	0.038			
	2nd	0.025	0.029	0.033	0.038	0.040	0.054
after	2nd	0.046					

Table 2. Thermal diffusivity of an amorphous metal ribon of $Ni_{78}Si_8B_{14}$.

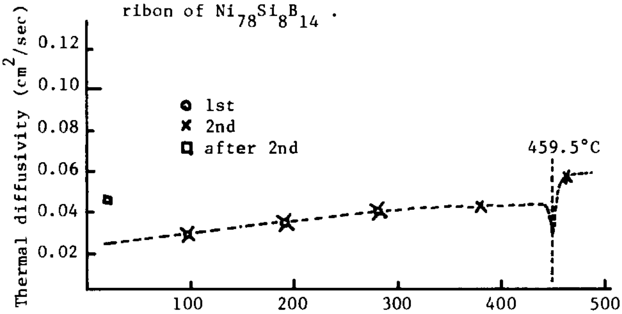

Fig.4. Thermal diffusivity of an amorphous metal ribon of $Ni_{78}Si_8B_{14}$

Temperature(°C)		24	95	193	290	386	428	472
Thermal diffusivity	1st	0.202	0.191	0.161	0.137	0.126	0.141	0.145
	2nd	0.200		0.159	0.139			0.146
	TPRC	0.229	0.198	0.168	0.136	0.123	0.142	0.145

Table 3. Thermal diffusivity of nickel.

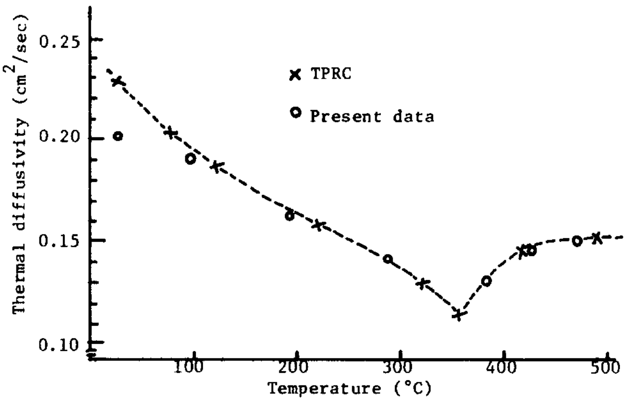

Fig.5. Thermal diffusivity of nickel.