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### Multifunctional Thermal Diffusivity and Thermal Conductivity Measuring Equipment: Thermowave Analyzer Measuring Principle and Equipment

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#### 1. Introduction

Since recent smartphones and tablet PCs have thinner housings and improved CPU performance, the challenge is how to dissipate the heat generated. Graphite sheets, which are thin and have high thermal conductivity, are used as heat-conductive materials.

In addition, with the increasing electrification of automobiles, thermal design to efficiently dissipate the large amount of heat generated by power devices such as inverters is becoming increasingly important. Thermal interface materials are used to reduce thermal resistance when dissipating the heat generated by power devices.

This document describes the principle and device for measuring thermal diffusivity of graphite sheets and thermal interface materials by periodic spot heating and radiation thermometry, a type of periodic heating method.

This measurement method combines periodic heating by laser and temperature measurement by radiation thermometer. This method is characterized by the fact that it does not require a heater for heating and a thermocouple for temperature measurement, thus allowing non-contact measurement of thermal diffusivity.

It is also possible to measure the thermal diffusivity in out of plane and in-plane directions of the sample and to map the thermal diffusivity in out of plane direction, making it possible to measure samples with high thermal diffusivity in the in-plane direction, such as graphite sheets, and samples with anisotropic and non-uniform properties, such as TIM. [1]

2. Principle of periodic spot heating-radiation thermometry

A sample surface with thermal diffusivity  $\kappa$  is spot heated by a periodic heating source  $P_{0}e^{i\omega t}$ . The alternating component of temperature at the heating point is expressed as  $T_{ac}=T_{0}e^{i\omega t}$ . The temperature propagation induced by the periodic heating source  $P_{0}e^{i\omega t}$  can be expressed by the following equation.

$$T_{\rm ac} = \frac{P_0}{4\pi\kappa rc} \cdot e^{-kr+i(\omega t - kr)} \tag{1}$$

c is the specific heat capacity per unit volume, r is the distance from the point heat source, and k is the wavenumber of the temperature wave expressed as

$$k = \sqrt{\frac{\omega}{2\kappa}} = \sqrt{\frac{\pi f}{\kappa}} = \frac{1}{\mu}$$

 $\mu$  is the thermal diffusion length here. Therefore, the phase in equation (1) is given by

$$\theta = -\sqrt{\frac{\pi f}{\kappa}} \cdot r \qquad (2).$$

2.1 In-plane direction measurement (distance variation method)

Figures 1 and 2 show schematic diagrams of in-plane direction measurement (distance variation method).

Let *r* in equation (2) be the distance *I* from the heating point and plot the distance *I* on the horizontal axis and the phase  $\theta$  on the vertical axis. Since the slope of the resulting graph is  $a=-(\pi f/\kappa)^{0.5}$ , the thermal diffusivity is

$$\kappa = \pi f / a^2 \tag{3}$$

Find the change in phase with respect to the change in distance, derive the slope *a* from the relationship, and substitute it into the above equation (3) to obtain the thermal diffusivity  $\kappa$ .

2.2 Measurement in the out of plane direction (frequency variation method)

Figures 3 and 4 show schematic diagrams of the out of plane direction measurement (frequency variation method).

Unlike in-plane measurement, measurement is performed while the heating point and detection point are coaxial.

Let *r* in equation (2) be the sample thickness d, plot the square root of the frequency on the horizontal axis, and Since the slope of the resulting graph is  $a=-(\pi/\kappa)^{0.5}d$ , the thermal diffusivity  $\kappa$  is

$$\kappa = \pi d^2 / a^2 \tag{4}$$

Obtain the phase with respect to the frequency characteristics, derive the slope *a* from the relationship, and substitute this into the above equation (4) to obtain the thermal diffusivity  $\kappa$ .

Thermal diffusivity can also be mapped by moving only the sample while keeping the heating and detection points coaxial.



Radiometric Thermometry





Figure 2 Schematic diagram of measurement results for inplane direction measurement (distance variation method)



Periodic heating (frequency f) )

Figure 3 Schematic diagram of out of plane measurement (frequency variation method)



Figure 4 Schematic diagram of measurement results of out of plane measurement (frequency variation method)

#### 3. Measurement device

#### 3.1. Device Configuration

Figure 5 shows a block diagram of the device configuration for periodic spot heating-radiation thermometry. The laser is modulated with a sinusoidal wave of frequency f by a signal generated by a function generator and focused on the sample surface.

The periodic heating temperature modulation (sine wave) generated by the periodic heating of the sample surface propagates through the sample and is detected by a radiation thermometer at the back surface. The temperature modulation signal detected by the radiation thermometer is input to a lock-in amplifier, where the phase delay relative to the function generator signal is measured.

In the case of measurement in the out of plane direction, the frequency generated by the function generator is changed, and in the case of measurement in the in-plane direction, the phase of the heating modulation signal is measured while changing the position of the infrared detection optical system using a mechanical stage. The lock-in amplifier, function generator, and mechanical stage are automatically controlled by a PC to acquire data.

For in-plane direction thermal diffusivity measurement, the thermal diffusivity is obtained from the slope of the relationship between distance and phase, and for out of plane direction thermal diffusivity, from the slope of the relationship between the square root of frequency and phase. Two types of mechanical stages are mounted. The mechanical stage for in-plane direction measurement changes the heating point on the sample surface and the signal detection point on the sample backside to change the positions of the heating and detection points in 1  $\mu$ m increments.

When the heating and detection points are coaxial, thermal diffusivity is measured in the out of plane direction. In this condition, thermal diffusivity can be mapped by moving only the sample by a mechanical stage.



Figure 5: Block diagram of the periodic spot heating radiometry thermal diffusivity measurement system

#### 3.2. sample table

This measurement technique requires no sample fixation and no attachment of a heater or thermocouple to the sample. Figure 6 shows a photograph of the sample stand.

The laser is irradiated from above and the temperature change is detected from below by an infrared radiation thermometer. Figure 6a shows the sample table without a sample. The upper cylinder is the optical system for the laser.

The signal is detected through the lower aperture. Figure 6b shows the sample installed. The preparation for measurement is completed by simply placing the sample. Figure 6c shows the sample holder placed on top of the sample. The sample holder should be placed only when the sample is thin and undulating. By selecting the heating modulation frequency appropriately, the edge surfaces of the specimen base, specimen, and specimen holder will not be affected. This is because the wavelength of the periodic temperature change becomes shorter as the frequency is increased, and the amplitude of the periodic temperature change is almost completely attenuated by one wavelength. [2]

3.3. Thermowave Analyzer TA, periodic spot heating and radiation thermometry thermal diffusivity measurement system

It is sold as the Thermowave Analyzer TA, which is compatible with sheet and thin plate materials and enables measurement in out of plane (vertical) and inplane (horizontal) directions of a sample and thermophysical property mapping within the sample. Figure 7 shows the appearance of the Thermowave Analyzer TA. It has the following features (1) to (8) and is used for measurement in many fields such as graphite sheets, thermal interface materials, highly thermally conductive resins, and CFRP.



Fig. 6 a Sample table (without sample in place)



Fig. 6 b Sample table (with sample in place)



Fig. 6 c Sample table (with sample and sample holder in place)

Main Features of Thermowave Analyzer TA

(1) Both out of plane and in-plane directions can be measured on the same sample.

- (2) High degree of freedom in sample shape.
- (3) Relatively thin samples can be measured.
- (4) Distribution measurement (relative value) is possible,

and can be used for non-destructive testing, such as evaluation of adhesion and filler bias.

(5) The wide measurement range enables measurement of everything from polymer materials to diamond.

(6) Laser safety is FDA standardized and equivalent to JIS Class 1.

(7) CE Marking is obtained.

(8) Automatic laser focus adjustment.



Fig. 7 Bethel Thermowave Analyzer TA, periodic spot heating radiometry thermal diffusivity measurement system

4. thermal diffusivity measurement of certified reference materials

To verify the measurement results, samples with certified or calibrated values were measured [3]. The certified value of thermal diffusivity at 300 K is  $9.51 \times 10^{-6} m^2 s^{-1}$  (relative expanded uncertainty 6.1%, coverage factor k=2) for NMIJ CRM5807-a certified reference material of alumina titanium carbide (Al<sub>2</sub>O<sub>3</sub>-TiC) distributed by AIST [4]. Isotropic graphite with a thermal diffusivity calibration certificate valued by the National

#### 6. References

Institute of Advanced Industrial Science and Technology, with a thermal diffusivity of 92.5x10-6m2s-1 at 298 K (relative expanded uncertainty 4.6%, inclusion factor k=2) [5]. The measured results for alumina-titanium carbide and isotropic graphite agreed within  $\pm 5\%$  compared to certified or calibrated values as shown in Table 1.

Table 1 Measurement results of standard samples for verification

		Thermal diffusivity /x10 <sup>-</sup> <sup>6</sup> m²s <sup>-1</sup>	
Sample	Measurement		Certified
name	direction	Measurement	value or
		value	calibration
			value
Al <sub>2</sub> O <sub>3</sub> -TiC	Out of plane	9.79	9.51
	In plane	9.04	
Isotropic	Out of plane	91.8	92.5
Graphite	In plane	92.5	92.0

## Table 1 Measurement results of standard samples for verification

#### 5. Summary

The measurement principle of the thermowave analyzer, the device, and the results of measurements of standard samples were presented. This device has features not found in conventional devices, such as the ability to measure anisotropy and mapping of thermal diffusivity of a sample. In addition, the thermal conductivity can be obtained by separately providing the specific heat capacity and density. With the recent strong demand for energy conservation, this device is indispensable for obtaining accurate thermophysical property values and designing products with high precision.

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- [3] [3] H. Hatori, T. Awano, C. Kobayashi, K. Hayashi, K. Horie, G. Nishi, H. Ohta, Proceedings of the 39th Japan Symposium on Thermophysical Properties, (2018), E112
- [4] [4] Thermal Diffusivity Certified Material (NMIJ CRM5807-a) Datasheet, National Institute of Advanced Industrial Science and Technology (AIST), (2018), E112
- [5] [5] Hitoto Hatori, Tetsuya Otsuki, Takahiko Kubota, Makoto Sekine, Takashi Yagi, Megumi Akoshima, Proceedings of the 37th Japan Symposium on Thermophysical Properties, (2016), C311

\*The measurement results shown in this datasheet are typical results and do not guarantee individual measurement results.

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\*The product specifications described in this data sheet are subject to change without notice.

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