Micro-area thermophysical property measurement system: Measurement principle and equipment of the Thermal Microscope

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1. Introduction

Decarbonization has become an international goal to achieve a sustainable society. Electrification of automobiles and other sectors is essential for efficient use of energy. In some areas, conversion to heat-pump air conditioners is also necessary. Inverter control is essential to control these devices, but how to dissipate the heat generated is critical for efficient and stable control. For these heat dissipation measures, the materials used for substrates and packages are important, and ceramics are used as heat dissipation materials. Ceramics may be used as a single material or as a composite of resin and ceramics as filler to achieve high thermal conductivity of resin. In both cases, it is important to measure the microscopic thermal conductivity distribution of the ceramics and the thermal conductivity of the filler alone. Bulk materials of mm to several tens of mm in size are used for thermal conductivity measurement, and it has been exceedingly difficult to measure the thermal conductivity of minute portions.

This document introduces the measurement principle and measurement examples of the thermal microscope, which enables the measurement of thermal conductivity on the micrometer order by means of periodical heating with a laser and the thermo-reflectance method, which enables measurement of temperature changes in a small area. [1]

2. Principle of the periodical heating thermoreflectance method

The thermoreflectance method is a temperature measurement method that makes use of the fact that the reflectance of a material changes with temperature. The use of this method has been expanding in recent years as a temperature measurement method with high temporal and spatial resolution. The periodical heating method has long been used as a method for thermal analysis and thermal conductivity measurement. The thermal microscope measures the thermal conductivity of a small area using the periodical heating thermoreflectance method, which combines these methods.

The principle of the measurement is shown in Figure 1. A Mo film is deposited on the sample and the surface is periodically heated by a heating laser. Heat propagates from the Mo film to the sample, causing a phase delay in the temperature response of the sample surface. The phase delay varies depending on the thermal properties of the sample. Since the reflectance of Mo varies with temperature, the relative temperature change of the surface can be measured by measuring the intensity change of the detection laser irradiated coaxially with the heating laser.

When the sample surface is heated with a laser whose intensity is modulated by a sine wave with an angular frequency ω, the phase delay δ of the alternating current component of the surface temperature response is expressed by equation (1).

$$\delta = \frac{3}{4}\pi + arctan\left(\frac{cosh^2\sqrt{\frac{\omega\tau_f}{2}}\left(tanh\sqrt{\frac{\omega\tau_f}{2}}+\beta\right)\left(tanh\sqrt{\frac{\omega\tau_f}{2}}+\beta^{-1}\right)}{cos^2\sqrt{\frac{\omega\tau_f}{2}}(\beta-\beta^{-1})tan\sqrt{\frac{\omega\tau_f}{2}}}\right)$$

(1)

$$\tau_f = \frac{d^2}{\alpha_f} \quad (2)$$

$$\beta = \frac{b_s}{b_f} = \frac{\sqrt{\lambda_s\rho_s C_s}}{\sqrt{\lambda_f\rho_f C_s}} \quad (3)$$

Here τ_f is the characteristic time for the thermal diffusivity of the thin film, d is the thickness of the thin film, b is the thermal effusivity, λ is the thermal conductivity, ρ is the density, C is the specific heat, and the subscripts f and s are metal and sample, respectively. Therefore, by measuring the phase lag δ, the thermal effusivity b_s of the sample can be obtained. The thermal conductivity of the sample can also be obtained from the following equation.

$$b_s = \sqrt{\alpha_s}\rho_s C_s = \sqrt{\lambda_s\rho_s C_s}$$

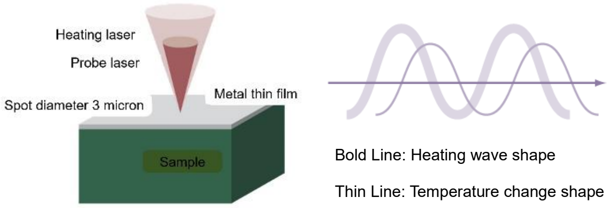

Fig. 1 Measurement principle

3. Measurement Device

Figure 2 shows the appearance of the TM3B thermal microscope. It has the following features (1) to (6) and is used for measurement in many fields such as various fillers, microstructures of ceramics, silicon thermal oxide films, etc.

Key features of the TM3B Thermal Microscope

(1) Minimum measurement resolution is 3µm.

(2) Capable of measuring minute areas.

(3) Capable of measuring submicron thin films.

(4) Thermal conductivity measurement of individual fillers is possible.

(5) Mapping measurement is possible.

(6) Measurement of only the surface layer of the sample is possible.

Figure 2 Bethel Periodical Heating Thermo-Reflectance Thermal Conductivity Measurement System
Thermal Microscope TM3B

4. Measurement example

4.1. Measurement example of functionally graded materials [1]

Figure 3 shows an example of measurement of functionally graded materials. This material is a sample of iron coated with an intermetallic compound of Al3Ti. By covering the metal surface with a heat-resistant and oxidation-resistant film, metal structural materials can be used at elevated temperatures. It also has a structure that alleviates thermal stress by changing the composition of the bonding surfaces of each material in a gradient manner. The cross section of the material was observed using a thermal microscope. Mapping measurements were conducted over an area of 140 µm x 190 µm and expressed as thermal effusivity.

Reasonable thermal effusivity was obtained for each of the iron phase and Al3Ti phase. At the boundary between each phase, the thermal effusivity gradually changes over about 20 µm, and the thermal effusivity changes with the composition.

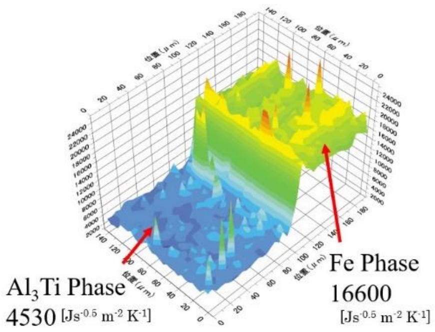

Figure 3 Mapping measurement of graded functional materials

4. 2. Example of SiC ceramics measurement [2]

Figure 4 shows an example of SiC ceramics measurement. A 40 µm x 40 µm area was mapped by measuring the thermal effusivity of the SiC ceramics. The thermal effusivity is not uniform, but rather distributed. Table 1 shows the thermal effusivity and thermal conductivity of diverse types of SiC. The single crystals have high thermal effusivity and thermal conductivity, while the polycrystals have low thermal effusivity and thermal conductivity.

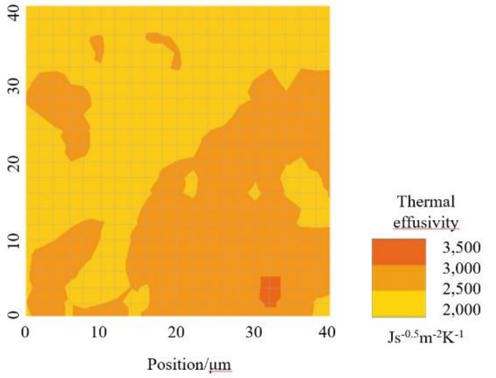

Figure 4 Mapping measurement of SiC ceramics

Table 1 Thermal Effusivity and Thermal Conductivity of Various Types of SiC

Material Name	Thermal diffusivity [Wm^{-1}K^{-1}]	Thermal effusivity [Js$^{-0.5}$m^{-2}K^{-1}]
4H-C SiC single crystal	440	30,400
6H-C SiC single crystal	366	28,100
SiC polycrystalline	274	24,200

*Values calculated as $\rho C_p=2.1\times10^6$[Jm^{-3}K^{-1}].

5. Summary

The measurement principles, equipment, and measurement results of the thermal microscope were shown. This device can measure thermal conductivity and mapping of microscopic areas of samples and has features not found in conventional devices. With the recent strong demand for energy conservation, this device is indispensable for obtaining accurate thermophysical property values and developing highly functional materials.

6. References

[1] K. Hatori, N. Taketoshi, T. Baba, H. Ohta, “Thermoreflectance technique to measure thermal effusivity distribution with high spatial

resolution,” Review of scientific instruments 76, 114901 (2005).

[2] Ikuko Yamada, Masaichi Kume, Koji Watari, Kimihito Hatori, Genzo Matsui, "Thermal Effusivity/Thermal Conductivity of Silicon Carbide Ceramics in Small Area", Thermophysical Properties 2008, Vol. 22 No. 3 p. 172-176

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