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Development of A Thermophysical Handy Tester for Non-Destructive Evaluation of Engineering Materials[1]

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Techniques of quality inspection and non-destructive diagnosis of engineering materials have been developed as procedures for determining various physical properties. The diagnostic techniques are usually based on detecting a very delicate signal concerned with changeable properties, and therefore detecting such a signal requires a great deal of experience in the procedures. *In situ* measurement for thermophysical properties of engineering parts is also important for judgment of the intrinsic soundness of the parts or for distinction between similar materials. The purpose of this study is the development of a thermophysical properties handy tester that can be used to simultaneously measure thermal conductivity and thermal effusivity and can be applied to the non-destructive diagnosis of numerous engineering materials. The tester consists of a portable data-acquisition unit, a probe holder equipped with a thermal probe, and a notebook computer. Measurements can be carried out by merely bringing the probe into point contact with a testing body for a period of 10 s. The thermal probe is constructed from a thin thermocouple in order to satisfy conditions based on the measurement principle. Materials that can be measured include polymeric resins, glasses, ceramics, alloys, and pure metals, among others.

KEY WORDS: comparative method; *in situ* measurement; point contact; solids; thermal conductivity; thermal effusivity.

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1. INTRODUCTION

Utility of information on thermophysical properties for the technique of quality inspection or distinction between similar materials has recently gained notice. Although a strict measurement method is important for determining accurate thermophysical properties, an easy measurement method is also necessary for *in situ* measurement, such as non-destructive quality inspection of engineering materials. Most existing methods of measuring thermophysical properties require some kinds of test pieces, and they cannot be applied to *in situ* measurement.

Measurements of the thermal conductivities of dielectric thin films by use of the thermal comparator method [1,2] have been reported. The comparator method is a candidate for *in situ* measurement, since it uses a point contact method. However, this method involves some problems: the contact pressure must be maintained constant, and more severe conditions are imposed on surface qualities, such as hardness.

A method of measuring three thermophysical parameters of solids by a thermal probe with instantaneous point contact [3,4] has recently been developed. This method is expected to find wide use in industry, because simple, easily accessible measurement is possible. Therefore, the purpose of this study is to construct a practical thermophysical properties handy tester based on this method in order to carry out *in situ* measurement. The newly developed tester has several advantages over the thermal comparator, not the least of which is an instantaneous method for measuring three thermophysical parameters (thermal conductivity, thermal diffusivity, and specific heat capacity) of solids. The tester consists of a portable data-acquisition unit having a dc power supply, a probe holder equipped with a thermal probe, and a notebook computer. Measurements can be carried out by merely bringing the probe into point contact with a testing body for a period of 10 s. The probe of this tester is composed of a thin Type-E thermocouple in order to satisfy measuring conditions. This tester does not require special test pieces, and testing bodies need not always have a plane surface as a result of the point-contact measurement. Measurement accuracy depends upon that of the test measurement for determining the thermal constants of the probe, since this is a comparative measurement. Materials that can be measured include polymeric resins, glasses, ceramics, alloys, and pure metals, among others. Typical applications are presented in this paper, such as the evaluation of the effective thermal conductivity of an alumina-FRP and inspection of chemical composition heterogeneity of an intermetallic compound alloy, NiAl-slab.

2. PRINCIPLE

Engineering solid materials are usually made of isotropic or orthotropic materials. Heat conduction of an orthotropic medium in the rectangular coordinate system was suggested for the first time by Carslaw and Jaeger[5] as follows:

$$(\rho c)_e \frac{\partial T}{\partial t} = \lambda_x \frac{\partial^2 T}{\partial x^2} + \lambda_y \frac{\partial^2 T}{\partial y^2} + \lambda_z \frac{\partial^2 T}{\partial z^2} \tag{1}$$

New independent variables X, Y, Z are defined as

$$X = \left(\frac{\lambda_e}{\lambda_x}\right)^{1/2} x, \quad Y = \left(\frac{\lambda_e}{\lambda_y}\right)^{1/2} y, \quad Z = \left(\frac{\lambda_e}{\lambda_z}\right)^{1/2} z \tag{2}$$

where

$$\lambda_e = \left(\lambda_x \lambda_y \lambda_z\right)^{1/3}. \tag{3}$$

Moreover, in the case of heat conduction by a point heat source on a semi-infinite body, the spherical coordinate system, which is set as $r = \left(X^2 + Y^2 + Z^2\right)^{1/2}$, is convenient. Therefore, Eq. (1) takes the form,

$$\frac{\partial T}{\partial t} = a_e \left(\frac{\partial^2 T}{\partial r^2} + \frac{2}{r}\frac{\partial T}{\partial r}\right) \tag{4}$$

where the effective thermal diffusivity is defined as

$$a_e = \lambda_e / (\rho c)_e \tag{5}$$

Now, suppose that a testing material is one of the parts of an industrial product and brought into point contact with the tip of a thermal probe. Here, the tip is a junction bead of a thermocouple entered in the probe and maintained higher than room temperature before contact. Then, we assume that heat flow occurs through a small contact area of hemispherical shape of radius r_0, and the heat conducted into the testing material is expressed as

$$Q = 2\pi r_0^2 \left[-\lambda_e \left(\frac{\partial T}{\partial r}\right)\right]_{r=r_0} \tag{6}$$

The contact surface of the thermal probe can also be assumed to be a small hemisphere of radius r_0, which is smaller than the tip of the probe. The transient temperature of the junction bead observed at the sensing

point r_1, which is quite close to the contact surface, is said to be the temperature response of the thermal probe just after contact. We obtain the analytical solution of the temperature response on the assumption that (1) both the tip of the probe and the testing material are considered to be semi-infinite from the viewpoint of a small area associated with such a small temperature change; (2) testing materials are opaque; (3) no heat effects and gas convection in material during experiment; and (4) the temperature dependence of thermophysical properties is negligible.

Heat conduction equations on the sides of the probe and the testing material are

$$\frac{\partial T_p}{\partial t} = a_p \left(\frac{\partial^2 T_p}{\partial r^2} + \frac{2}{r} \frac{\partial T_p}{\partial r} \right) \tag{7}$$

$$\frac{\partial T_s}{\partial t} = a_s \left(\frac{\partial^2 T_s}{\partial r^2} + \frac{2}{r} \frac{\partial T_s}{\partial r} \right) \tag{8}$$

where the thermal diffusivity of the probe is defined as $a_p = \lambda_p/(\rho c)_p$ and, instead of Eq. (5), that of the testing material is $a_s = \lambda_s/(\rho c)_s$.

Conditions of continuity for both temperature and heat flow on the contact surface are

$$T_p(r_0, t) = T_s(r_0, t) \tag{9}$$

$$\lambda_p \left. \frac{\partial T}{\partial r} \right|_{r=r_0} = -\lambda_s \left. \frac{\partial T}{\partial r} \right|_{r=r_0} \tag{10}$$

Initial and boundary conditions are set as

$$T_p(r, 0) = T_{p_0}, \quad T_s(r, 0) = T_{s_0} \tag{11}$$

$$T_p(\infty, t) = T_{p_0}, \quad T_s(\infty, t) = T_{s_0} \tag{12}$$

where T_{p_0} and T_{s_0} are initial temperatures and are uniform in each body, respectively.

The dimensionless temperature in the probe is defined as $T_p^* = (T_p - T_{p_0})/(T_{s_0} - T_{p_0})$.

By solving Eqs. (7)–(12), the analytical temperature response at $r = r_1$ is obtained as follows:

$$T_p^* = \frac{\beta}{\eta(\beta+1)} \operatorname{erfc}\left(\frac{C}{\sqrt{t}} \right) + \frac{\zeta - \beta}{\eta(\zeta+1)(\beta+1)} \exp\left(X^2 - \frac{C^2}{t} \right) \operatorname{erfc}(X) \tag{13}$$

where

$$\beta = \frac{\lambda_s}{\lambda_p}, \quad \zeta = \frac{\xi_s}{\xi_p}, \quad \eta = \frac{r_1}{r_0}, \quad C = \frac{r_1 - r_0}{2\sqrt{a_p}}, \quad X = \frac{C}{\sqrt{t}} + \frac{(\beta+1)(\eta-1)}{2C(\zeta+1)}\sqrt{t} \tag{14}$$

In the period satisfying $C\sqrt{t} \ll 1$, Eq. (13) can be expressed as

$$T_p^* \cong \frac{\beta}{\eta(\beta+1)} - \frac{b}{\eta(\beta+1)}\left\{\beta - \frac{\zeta-\beta}{(\eta-1)(\beta+1)}\right\}\frac{1}{\sqrt{t}} \tag{15}$$

where $b = 2C/\sqrt{\pi}$.

This implies that the temperature response T_p^* is approximated by a linear function of $1/\sqrt{t}$ in the latter period. Thus, the following relation can be applied to measured temperature responses in the similar period. That is,

$$T_p^* = A - B\frac{1}{\sqrt{t}} \tag{16}$$

If parameters A and B in Eq. (16) can be found by least squares and the values of both b and η can be determined, the thermal conductivity ratio β and the thermal effusivity ratio ζ can be calculated from Eqs. (15) and (16) as follows:

$$\beta = \frac{A\eta}{1 - A\eta} \tag{17}$$

$$\zeta = (\beta+1)^2\left(A - \frac{B}{b}\right)\eta(\eta-1) + \beta \tag{18}$$

The procedure for determining the values of b and η has been discussed in a previous paper [3]. This procedure requires a lot of calculation time. Therefore, using a fixed value of b, we can determine the value of η by curve fitting the analytical temperature response, Eq. (13), with the measured one. The reason why b can be fixed is that the distance between the thermal sensing point of the probe and its contact point with a testing material is constant, so long as the same probe is used.

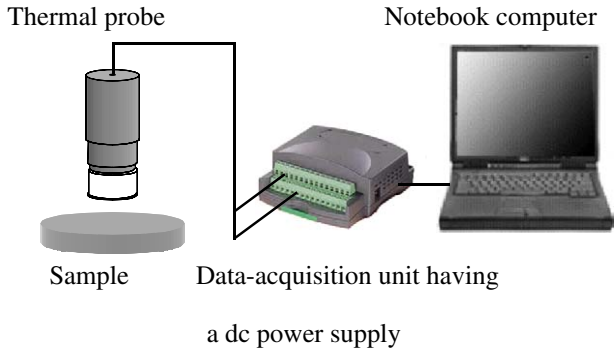

Fig. 1. Measuring system of the thermophysical handy tester.

3. MEASUREMENT SYSTEM

Figure 1 schematically shows the newly developed thermophysical handy tester. The tester consists of a portable data-acquisition unit having a dc power supply, a probe holder equipped with a thermal probe, and a notebook computer. Figure 2 shows the thermal probe within the probe holder. The overall length of the holder is about 120 mm. The probe holder can be manipulated in one hand. The thermal probe is constructed from a Type-E ther-

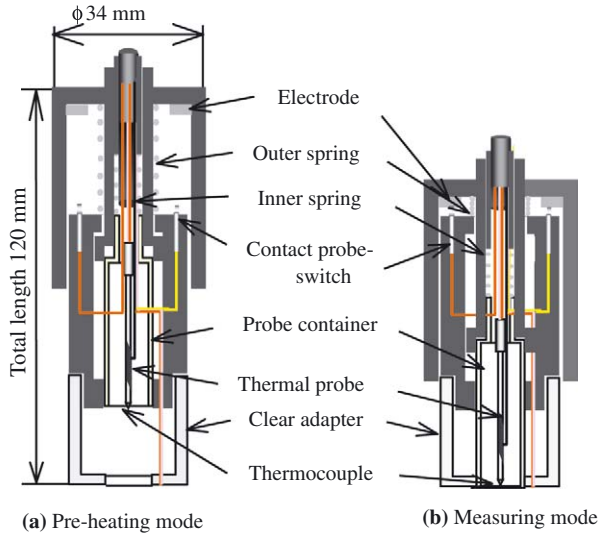

Fig. 2. Cross-sectional view of the probe holder.

mocouple with a wire of 0.1 mm diameter sheathed in an alumina tube of 0.8 mm diameter. The probe wrapped in a polystyrene-foam insulation is maintained at a temperature about 20 K above room temperature, by use of a small heater installed in it. The measurement can be carried out by merely bringing the probe into point contact with a testing body for a period of 10 s. Then, the tip of the probe is extruded by depressing the holder from the opposite side. In order to reduce thermal resistance on the contact surface, contact pressure is exerted by an inner spring to be about 10 MPa. The temperature response is measured at sampling intervals of 0.1 s. In order to start measuring just at contact, the trigger system is used by the aid of a contact probe-switch built into the holder. The temperature responses are measured with a portable data-acquisition unit. Subsequently, the data are converted into a CSV file and processed by use of the macro-function of Excel on a notebook computer. As a result, the temperature response curve is drawn on the display along with the result of the thermophysical properties calculated from the response.

4. MEASURED TEMPERATURE RESPONSES

Example temperature responses for some materials are plotted in Fig. 3. The thermophysical properties of these materials are known. As the figure shows, the response curves for many kinds of materials can be clearly distinguished. These response curves are characterized according to four parameters, β, ζ, η, and b. This measurement method evaluates the bulk properties in the vicinity of the point-contact surface to the depth of several millimeters. The value of η is determined by the size of the contact area in every measurement operation. In contrast, the distance between the contact point and the thermal sensing point of the probe does not

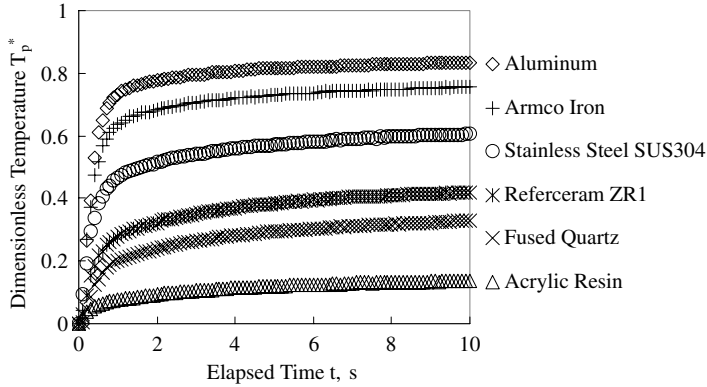

Fig. 3. Measured temperature responses for various materials.

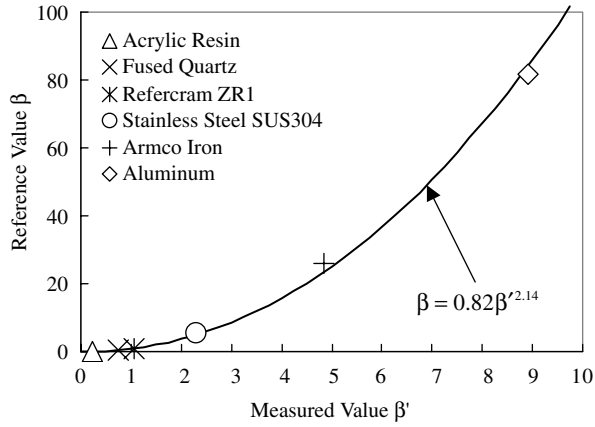

Fig. 4. Correlation of β between reference and measured values.

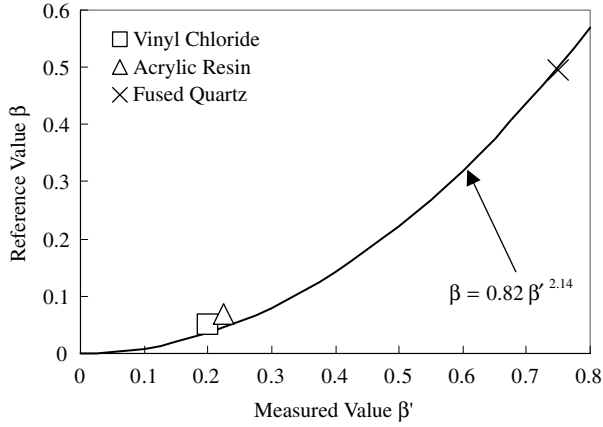

Fig. 5. Correlation of β between reference and measured values for materials of low thermal conductivity.

vary, if the tip of the probe is not transformed. Therefore, the value of b is constant so long as the same probe is used. The radius of the contact area of the probe may be less than 0.2 mm, in which case η is less than 1.1. In the analysis of these response curves, the value of b was determined as $b = 0.0252\,\mathrm{s}^{0.5}$ for this probe. Then, $C = 0.0224\,\mathrm{s}^{0.5}$. All the response curves were determined to become rectilinear for $1/\sqrt{t}$ after a period of 5 s.

Figure 4 shows the correlation of β between measured and reference values. The thermal conductivity of the probe was estimated from measurement of austenitic stainless-steel, SUS304. For the case of materials of

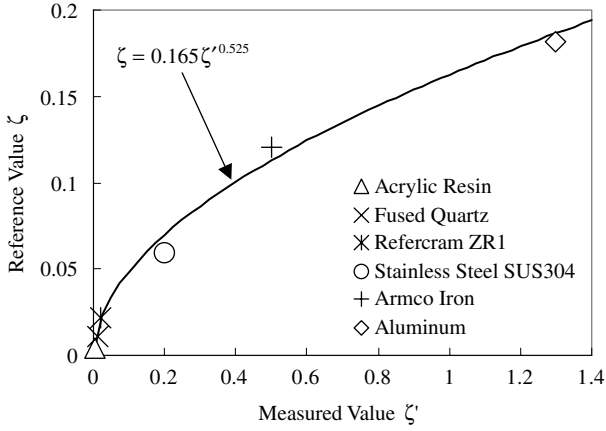

Fig. 6. Correlation of ζ between reference and measured values.

low thermal conductivity, the response is very reproducible and easily analyzed, as shown in Fig. 5, making this method useful for distinguishing polymeric resins. A similar comparison for ζ is shown in Fig. 6.

The relation between measured and reference values; that is, the correction equations obtained by the least-squares method, is presented in each figure. A measured response curve has a systematic error, because of slow response of a data-acquisition unit or practically unsatisfied conditions in the measurement principle, and thus a correction must be applied to the measured values of both β' and ζ'. If both β and ζ are obtained, the thermophysical properties, λ_s, ξ_s of a testing body are known from those of the probe; i.e. for this probe, $\lambda_p = 9.34\,\mathrm{W \cdot m^{-1} \cdot K^{-1}}$, $\xi_p = 29.1\ \mathrm{kJ \cdot m^{-2} \cdot s^{-0.5} \cdot K^{-1}}$.

5. APPLICATIONS

Composite materials such as fiber reinforced plastics and intermetallic compound alloys have been used in industrial products. The former are artificially made so as to be anisotropic. In contrast, the latter are required to be homogeneous. The thermophysical handy tester has been applied to the evaluation of thermal conductivity and the quality inspection of such materials.

Figure 7 shows the shape and coordinate system of a sample of an alumina-fiber reinforced plastic. The diameter of the fiber is about 10 μm, and the content ratio of the fiber is 70 vol%. The sample is formed into a cubic die measuring 10 mm by superimposing boards of 1 mm thickness.

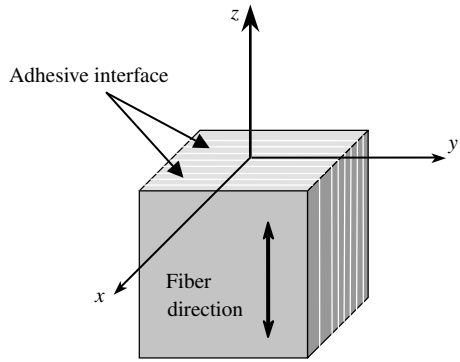

Fig. 7. Testing body of alumina-FRP and coordinate system.

It is obvious from the simulation shown in Fig. 8 that in the case of rather large contact area, the configuration of the temperature profile in the sample varies according to the direction of the fiber. As is obvious from Fig. 9, the temperature response measured in the direction of the z-axis is greater than those in the other directions, and the response curves in the directions of the x and y-axes agree with each other. Therefore, an important factor is that the diameter of contact area in this case is too large to be considered as a point, while the diameter of a fiber is sufficiently smaller than that of the contact area of the probe. From this result we also ascertain that the effective thermal conductivity λ_e evaluated in the direction of the x-axis is equal to that in the direction of the y-axis, because $\lambda_x = \lambda_y$ in Eq. (3). The effective thermal conductivity λ_e was obtained as $1.2\,\mathrm{W \cdot m^{-1} \cdot K^{-1}}$ in the direction of the z-axis.

A slab of an intermetallic compound alloy, NiAl, has been produced for a sputtering substrate. The slab measurement of 15 mm thickness and 129 mm radius, and is shown in Fig. 10 along with some measuring sites. The existence of the radial distribution of thermal conductivity in the slab was revealed as shown in Fig. 11, while the distribution in the circumferential direction was not detected. Such variation of thermal conductivity may be caused by the size of the grain boundary. The size depends not only on the relative proportion of the two components, Ni and Al, but also on the cooling speed upon casting. Consequently, this handy tester is useful for improving metallurgical processing of products, and is expected to find wide use in industry, by virtue of realizing an *in situ* measurement method.

6. SUMMARY

The handy tester described herein has been developed in order to apply thermophysical properties measurement to the techniques of quality

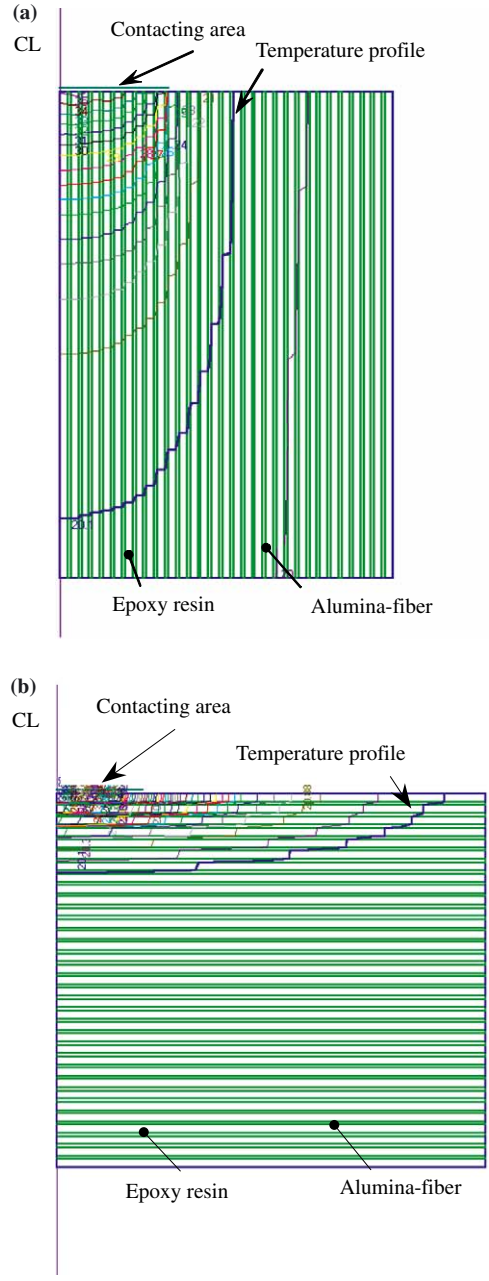

Fig. 8. Simulation of temperature profiles in the condition of the probe contacting (a) parallel to the direction of the fiber and (b) perpendicular to the direction of the fiber.

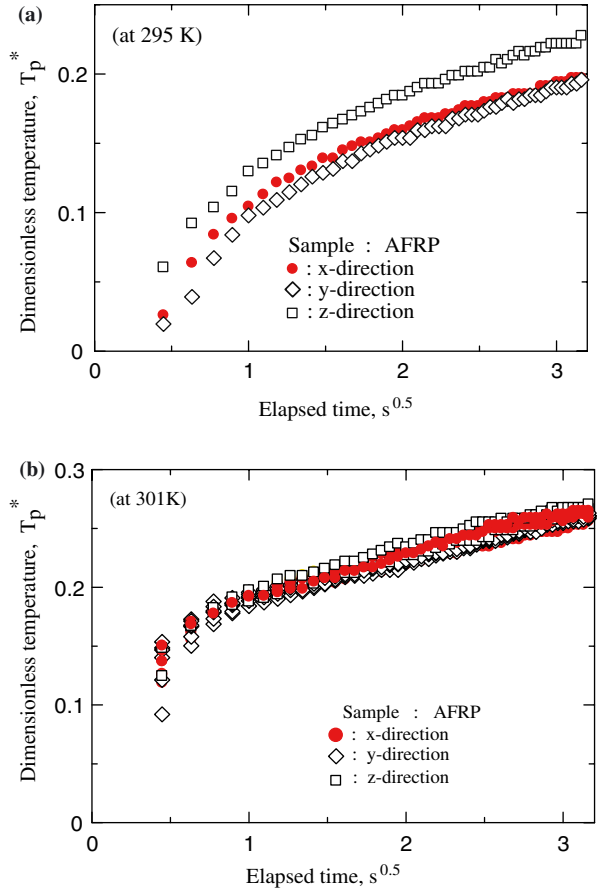

Fig. 9. Measured temperature responses for the alumina-FRP; (a) using the tip of 0.3 mm diameter and (b) using the tip of 0.1 mm diameter.

inspection and non-destructive evaluation. First, several kinds of known materials were measured in order to confirm operation of the tester by comparison with literature values. The obtained reproducibility of temperature response means that the tester can be used to measure thermophysical properties without influence of the contact radius when using a thin thermal probe. Moreover, the influence of convection around the tip of the probe was found to be of small significance. The temperature dependence of the thermophysical properties of materials can be sufficiently disregarded because of small temperature changes. As a result, the

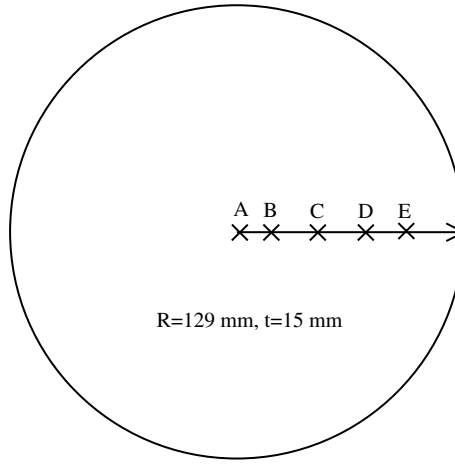

Fig. 10. NiAl slab for a spattering base-plate.

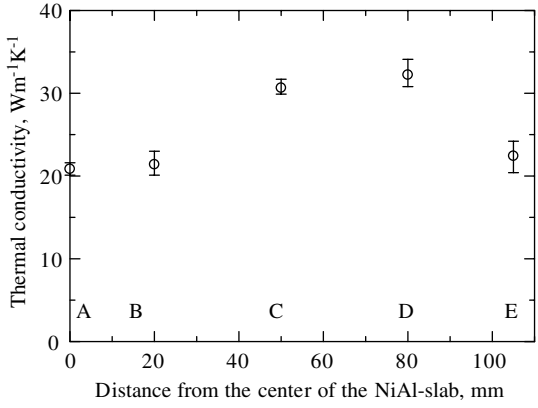

Fig. 11. Radial distribution of thermal conductivity in the NiAl slab.

accuracy of the measured thermophysical properties depends mainly on that of the probe constants determined by use of some standard materials. This method allows measurements for transparent materials such as fused quartz and for composite materials such as fiber-reinforced plastics. Although applicable materials cover the range from nonmetallic to metallic, this method cannot be applied for materials during phase and structure changes as ice melting, and for rubber-like or highly porous materials. This measurement method is expected to be used to develop technology

which can determine the existence of a micro-crack beneath the surface of a testing body or the degree of degradation of engineering solid materials.

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