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Determinaton of Thermal Diffusivity of Two-Layer Composites by Flash Method (Report 2)†
— Estimation of Accuracy in Measurement —

Katsunori INOUE* and Etsuji OHMURA**

Abstract

Accuracy in determining by the flash method proposed by the authors of the thermal diffusivity of one layer or two layer composites is estimated theoretically and experimentally. The present method can be applied to the two-layer composites with the thermal resistance at the interface of the layers, if the thermal conductivities of the two layers and the thermal diffusivity of the other layer are known. Influence of each relative error of thickness of layers, thermal conductivities, the other thermal diffusivity, and thirty- and seventy-percent time on accuracy of the results obtained by this method of thermal diffusivity and thermal resistance is estimated theoretically. The present method is applied to some joining specimens whose thermophysical properties have been all measured in advance by the laser flash half-time method. Influence of the amount of scatter in measured values of thirty- and seventy-percent time and influence of the combinations of various percent time on the estimated results of thermal diffusivity and thermal resistance are investigated. It is confirmed that the most important factor of accuracy in the present method is the measured time in temperature history of the rear surface such as thirty- and seventy-percent time. Influence of the accuracy of the thermophysical properties used on accuracy of the results is relatively small.

KEY WORDS: (Thermophysical Property) (Laser Flash Method) (Two-Layer Composite) (Thermal Diffusivity) (Thermal Resistance) (Accuracy)

1. Introduction

It is important to estimate quantitatively not only the bonding strength but also the thermophysical properties or characteristics of heat transfer of the various layer composites, especially ceramics joined with metals, coating materials on substrates and so on. The authors1) proposed a flash method of determining of the thermal diffusivity of two-layer composites. This method can be applied to the two-layer composites with the thermal resistance at the interlayer surfaces, if the thermal conductivities of the two layers and the thermal diffusivity of the other layer are known. The time required for the temperature to rise 30 and 70 percents, for example, of the maximum temperature increase is read from the temperature versus time curve obtained experimentally. These results are used for the numerical computation based on the theoretical equation of the normalized temperature increase on the rear face and the method of bisection algorithm. The thermal resistance at the interlayer surface can be obtained easily as well as the thermal diffusivity.

In this paper, accuracy in the determination of the thermal diffusivity by the present method is estimated theoretically and experimentally. Influence of each relative error of thickness of layers, thermal conductivities, the other thermal diffusivity, and thirty- and seventy-percent time on accuracy of thermal diffusivity and thermal resistance obtained by this method is estimated theoretically. The present method is applied to some joining specimens whose thermophysical properties have been all measured in advance by the laser flash half-time method2). Influence of the amount of scatter in measured values of thirty- and seventy-percent time and influence of the combinations of various percent time on the estimated results of thermal diffusivity and thermal resistance are also investigated.

2. Theory of Measurement

The schematic diagram of the geometry of a two-layer composite is shown in Fig. 1. In the flash method, the

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front surface of the sample is subjected to a short radiant energy pulse and the resulting temperature history of the rear face is recorded. If we set up the following assumptions are made:

1. One dimensional heat flow,
2. No heat loss from the sample surfaces,
3. Each layer is homogeneous,
4. Thermal contact resistance between layers is uniform,
and 5. Heat pulse is uniformly absorbed on the front surface.

The normalized temperature of the rear surface is represented as Eq. (1):

\[ \Theta = 1 + 4(\lambda^*d^* + \gamma^2) \times \sum_{n=1}^{\infty} \frac{e^{-\beta_n \gamma^2 t^*}}{(\lambda^*+\gamma)(d^*+\gamma) \cos \beta_n(d^*+\gamma) + (\lambda^* - \gamma)(d^*-\gamma) \cos \beta_n(d^*-\gamma)} \]

where

\[ \Theta = (1 + \frac{\lambda_1 d_1}{\lambda_2 d_2}) \frac{\lambda_2}{\lambda_1} \frac{d_2}{d_1} T_1(d_1, t), \]

\[ \gamma = \sqrt{\frac{a_1}{a_2}}, \lambda^* = \frac{\lambda_1}{\lambda_2}, d^* = \frac{d_1}{d_2}, R^* = \frac{\lambda_2 R}{\lambda_1}, \]

\[ t^* = \frac{a_2 t}{d_2^2}, \]

and \( T, a, \lambda, R \) and \( Q \) are the temperature increase, the thermal diffusivity, the thermal conductivity, the thermal resistance at the interlayer surface and the heat input per unit area on the front surface, respectively. The subscripts 1 and 2 mean the properties of each layer. The \( \beta_n \) is the \( n \)-th positive root of

\[ (\lambda^* + \lambda) \sin \beta(d^* + \gamma) + (\lambda^* - \gamma) \sin \beta(d^* - \gamma) + \beta\gamma \lambda^* R^*[\cos \beta(d^* + \gamma) - \cos \beta(d^* - \gamma)] = 0. \]

Postulating that the thermal diffusivity of the layer 1 and the thermal resistance at the interlayer surface in Fig. 1 are unknown, \( \gamma \) and \( R^* \) are unknowns and \( t^* \) is a variable in Eq. (1), then we can represent \( \Theta \) in the following form:

\[ \Theta = \theta(\gamma, R^*; t^*). \]

\( \theta \) is a monotone increasing function of \( t^* \) and converges to one when \( t^* \rightarrow \infty \), independently of the thermophysical properties and the thermal resistance of the two-layer composite. We introduce a new parameter \( t_\alpha^* \) defined by the following equation:

\[ \theta(t, R^*; t_\alpha^*) = \alpha, \]

where \( 0 < \alpha < 1 \).

When \( t_\alpha^* \) is known and \( \gamma \) and \( R^* \) are variables, the combination of \( \gamma \) and \( R^* \) which satisfies Eq. (5) is expressed as a curved line in the \( \gamma-R^* \) plane 

for one value of \( t_\alpha^* \), and these curved lines for various values of \( t_\alpha^* \) cross at a point, which shows the values of \( \gamma \) and \( R^* \) to be obtained. Therefore, if we express \( \gamma \) which satisfies Eq. (5) for any \( R^* \) like as \( \gamma(R^*; t_\alpha^*) \), both \( \gamma \) and \( R^* \) can be determined by solving the equation

\[ \gamma(R^*; t_\alpha^*) - \gamma(R^*; t_\alpha^*) = 0, \]

which holds for the two \( \alpha \)-values, \( \alpha_1 \) and \( \alpha_2 \). This solution can be obtained easily through the numerical calculation. In this study, the method of bisection algorithm was adopted.

3. Theoretical Estimation on Accuracy

Since the present method is an indirect measurement method, it is important to investigate the influence of the error of each parameter, that is, the thickness of each layer, the measured time and the thermophysical properties on the estimated results. If the values used for calculation, which are denoted by the superscript "'" , have relative error \( e \) for the true values, \( d_i, \lambda_j, a_2 \) and \( t_{i,k} (i, j, k = 1, 2) \), we can express these parameters are

\[ d_i' = (1+e_{d_i})d_i, \quad i = 1, 2 \]

\[ \lambda_j' = (1+e_{\lambda_j})\lambda_j, \quad j = 1, 2 \]

\[ a_2' = (1+e_{a_2})a_2, \]

\[ t_{i,k}' = (1+e_{t_{i,k}})t_{i,k}, \quad k = 1, 2. \]

Substituting Eq. (7) into Eq. (2), the dimensionless values of each parameter can be written as

\[ d_i'' = \frac{1+e_{d_i}}{1+e_{d_i}} d_i', \]

\[ \lambda_j'' = \frac{1+e_{\lambda_j}}{1+e_{\lambda_j}} \lambda_j', \]

\[ t_{i,k}'' = \frac{(1+e_{t_{i,k}})(1+e_{t_{i,k}})}{(1+e_{t_{i,k}})^2} t_{i,k}', \quad k = 1, 2. \]
The values of $\gamma$ and $R^*$ obtained through the present measuring method using the above values have relative errors $\varepsilon_\gamma$ and $\varepsilon_{R^*}$, and they are written as follows:

$$
\begin{align*}
\gamma' &= (1+\varepsilon_\gamma)\gamma, \\
R^* &= (1+\varepsilon_{R^*})R^*,
\end{align*}
$$

(9)

Therefore, $a_1'$ and $R'$ are expressed by

$$
\begin{align*}
a_1' &= (1+\varepsilon_{a_1})^2(1+\varepsilon_{\alpha_1})a_1, \\
R' &= \frac{(1+a_d')(1+\varepsilon_{R^*})R}{1+\varepsilon_{\lambda_2}}
\end{align*}
$$

(10)

from Eqs. (2) and (9), and the relative errors of $a_1'$ and $R'$ can be estimated by

$$
\begin{align*}
\varepsilon_{a_1} &= (1+\varepsilon_\gamma)^2(1+\varepsilon_{\alpha_1}) - 1, \\
\varepsilon_R &= \frac{(1+\varepsilon_{a_d})(1+\varepsilon_{R^*})}{1+\varepsilon_{\lambda_2}} - 1.
\end{align*}
$$

(11)

(12)

An example of the estimated results of errors are shown in Fig. 2. The values of each parameter used for calculation are shown in the box in the figure. The values of $\alpha_1$ and $\alpha_2$ are set to 0.3 and 0.7, respectively. The abscissa means the absolute value of the relative errors in Eq. (7), and the influence of each error of these parameters on $\varepsilon_{a_1}$ and $\varepsilon_R$ is shown in this figure. Either $\varepsilon^+$ or $\varepsilon^-$ mean that the error of each parameter is positive or negative, respectively, and the absolute values are equal each other, when other parameters have no error. It is clear that the values of $\varepsilon_{a_1}$ and $\varepsilon_R$ increase as the relative error of each parameter increases. Especially, when $\varepsilon_{\lambda_{0.3}}$ and $\varepsilon_{\lambda_{0.7}}$ have different sign, the relative errors of the estimated values become largest, and the errors of thickness are followed. The errors of thermal conductivities $\lambda_i$ ($i = 1, 2$) and thermal diffusivity $a_2$ do not affect the final accuracy so much.

4. Experiments

The experiments were carried out with a flash type thermal constant analyzer, TC 3000 HNC, developed by Sinku-Riko Inc. The apparatus consists of a ruby laser, a vacuum system with a sample holder and a heater, temperature detectors and a data acquisition system. The pulse width of the laser was about 0.7 ms. The temperature detector used was a radiation thermometer with an InSb sensor, and the temperature history of the rear face of the sample was recorded by a transient memory and a pen recorder.

We used two kinds of materials for specimens, that is, carbon steel for machine structural use, S45C, and stainless steel, SUS 304, which are equivalent to AISI 1045 and AISI 304, respectively. The thermpophysical properties of these materials are influenced by the contents. Therefore, the thermal diffusivity $a$ and the heat capacity $c_p$ of these materials were measured by the same apparatus using the conventional half-time method of the laser flash technique for a homogeneous layer and the laser-flash calorimetric method, respectively, then the thermal conductivity was calculated using the relation of $\lambda = (pc)a$. The measured results are shown in Table 1.

![Figure 2](image_url)  
Fig. 2 Influence of each relative error of $d_0$, $\lambda_{d_0}$, and $\lambda_{d_0.7}$ on accuracy of $a_1$, and $R$ obtained.

<table>
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<th>Material</th>
<th>$a$ m²/s</th>
<th>$\lambda$ W/(m·K)</th>
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<tr>
<td>S45C</td>
<td>$1.10\times10^{-3}$</td>
<td>38.2</td>
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<tr>
<td>SUS304</td>
<td>$3.55\times10^{-3}$</td>
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These properties will be used as the known values of $\lambda_1$, $\lambda_2$ and $a_1$ in the present method, and the thermal diffusivity will be also compared with the measured results of $a_1$ later on.

The shape of the specimens was a disk whose diameter was 10 mm and the thickness is from 0.7 to 0.8 mm. Both plane surfaces of the disk were finished finely by a lathe in order to get parallel planes. Spot welding and linking by silicon grease, which we call silicon linking, were used for joining two layers. A resistance welding machine, YR-500 CM, which was developed by Matsushita Industrial Equipment Co., Ltd., was used for the spot welding, and the welding force, the welding current and the weld time were set at $3.04 \times 10^5$ Pa, 13.5 kA and 133 ms, respectively. No deformation of the specimens through the spot welding was recognized. They were sectioned at the center line and vertically to the plane surface after the measuring experiments, and the transverse cross-section was observed by a metallographic microscope. It was confirmed that the two layers were almost uniformly joined at the interlayer surfaces.

5. Experimental Results and Discussions

In the present experiment, $a_1$ and $a_2$ in Eq.(6) were selected at 0.3 and 0.7, respectively, and the amount of scatter in measured value of $t_{0.3}$ and $t_{0.7}$ was investigated in advance, considering the results of theoretical estimation on accuracy obtained in section 3. The specimens were reset after the ruby laser was irradiated on them five times a setting, and the resulting temperature histories of the rear faces were recorded thirty times in total. The times $t_{0.3}$ and $t_{0.7}$ read from the thirty temperature-versus-time-curves are shown in Fig. 3 as a scatter diagram. The six symbols in the figure mean that the values of same symbol were obtained in the same setting. It is obvious from this figure that the deviation of measured data depends on the setting of specimens, whose reason has not been clear yet. The twenty-seven points plotted in Fig. 3, except each

![Fig. 3 Scatter in measured values of $t_{0.3}$ and $t_{0.7}$.](image)

![Fig. 4 Measured results of $a_1$ and $R$ with $t_{0.3}$ and $t_{0.7}$ shown in Fig. 3.](image)

one point of second-, third- and fifth-setting, lie almost in a straight line, therefore, an adequate correlation is estimated between $t_{0.3}$ and $t_{0.7}$. In Fig. 3, the mean values of these twenty-seven points and all of the thirty points are shown for reference by the symbols $\diamond$ and $\bullet$, respectively, and the standard deviation of them are also shown by symbol $I$. Both of the amounts of scatter of $t_{0.3}$ and $t_{0.7}$ are about 3.8%, and there is almost no difference between the twenty-seven points and all of the thirty points.

The thermal diffusivity $a_1$ of the layer 1 and the thermal resistance $R$ at the interlayer surface were obtained by the present method using the data in Fig. 3 and the thermophysical properties shown in Table 1 and the results are shown in Fig. 4. The six symbols in the figure are the same one in Fig. 3. The each one point of second-, third- and fifth-setting, which deviates from the straight line in Fig. 3, is an isolated point, respectively, as shown in Fig. 4. The mean values of these twenty-seven points and all of the thirty points are also shown by the symbols $\diamond$ and $\bullet$, respectively, and the standard deviation of them are shown by symbol $I$. The value of $a_1$ in Table 1 is shown by a broken line for reference. The relative errors of the mean value of $a_1$ in Fig. 4 to the known value in Table 1 are 0.45% and 6.0% for the twenty-seven points and all of the thirty points, respectively. The amounts of scatter of $a_1$ and $R$ for all of the thirty points are 17.2% and 28.1%, respectively. On the other hand, the amounts of scatter of them for the twenty-seven points are 6.1% and 11.9%, respectively, which indicate that the values are concentrated on the mean value relatively well.

The five points of the first setting, which are shown by the symbol $\varnothing$, are the another group separated from the rest in Fig. 3, but they are closer to the rest in Fig. 4. Therefore, it is concluded that if the amounts of scatter of the time $t_{0.3}$ and $t_{0.7}$ are smaller with the same degree,
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method can be also applied for any $\alpha_1$ and $\alpha_2$, theoretically, therefore the thermal diffusivity and the thermal resistance at the interlayer surface for both of the silicon-linked composites of S45C-S45C and S45C-SUS304 were also measured for other combinations of the times $t_{\alpha_1}$ and $t_{\alpha_2}$. The results are shown in Fig. 5, where the times of $t_{0.1}$, $t_{0.2}$, ..., $t_{0.9}$ were read from the each temperature versus time curves, and $a_1$ and $R$ are measured for the every combinations of them. The abscissa shows the values of which correspond to $t_{\alpha}$ and the absolute of the difference between $\alpha_2$ and $\alpha_1$. The circles in the figures mean the values of $\alpha_1$, which are shown in the tables under the figures. As seen from Fig. 5, the amount of scatter in measured values of $a_1$ and $R$ is larger as the difference between $\alpha_1$ and $\alpha_2$ is smaller. This tendency can be observed more remarkably for the silicon-linked composites of S45C-S45C. This result can be understood easily, considering that the well distant two-points on the temperature versus time curve show the characteristics of the curve more clearly. The drastic increase of temperature at the laser irradiating time causes error in measuring temperature, on the other hand, it is difficult to read the time precisely near the maximum temperature point on the temperature versus time curve, because the temperature increases gently near the point. It is concluded that it is proper to select the values of about 0.3 and 0.7 for $\alpha_1$ and $\alpha_2$, respectively.

6. Conclusions

Accuracy in determining by the flash method proposed by the authors of the thermal diffusivity of one layer of two layer composites is estimated theoretically and experimentally. The results obtained can be summarized as follows:

(1) The most important factor of accuracy in the present method is the times $t_{\alpha_1}$ and $t_{\alpha_2}$ read from the temperature versus time curve of the rear surface. If the amount of scatter of the time $t_{\alpha_1}$ and $t_{\alpha_2}$ are smaller with the same sign, the relative errors of $a_1$ and $R$ are smaller.

(2) The amount of scatter in measured values of $a_1$ and $R$ was larger as the difference between $\alpha_1$ and $\alpha_2$ is smaller. It is proper to select the values of about 0.3 and 0.7 for $\alpha_1$ and $\alpha_2$, respectively.

(3) Influence of the accuracy of the thermophysical properties used on accuracy of the final results is relatively small.

(4) The influence of the parallelism of each layer of the composites and the uniformity of the absorbed energy density on the accuracy of the measured results is the subject for a future study.
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