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Determination of Thermal Diffusivity of Two-Layer Composites by Flash Method†

Katsunori INOUE* and Etsuji OHMURA**

Abstract

A laser flash method of determining the thermal diffusivity of one layer of two-layer composites with the thermal contact resistance at the interlayer surfaces is described. A flash pulse is absorbed in the front surface of a thermally insulated specimen, and the resulting temperature history of the rear face is measured by a radiation thermometer and recorded with a transient memory and a pen-recorder. 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. 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, and the thermal contact resistance can be obtained easily as well as the thermal diffusivity. The fundamental equation of the temperature increase on the rear face in the present one-dimensional heat conduction problem is derived by Laplace transformation. 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, and the effectiveness of this method is confirmed. The thermal contact resistance is also measured in addition by another method already proposed by the authors, and these obtained results are in fairly good agreement each other.

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

1. Introduction

Recently, various layer composites, especially joining ceramics with metals or coating materials on substrates, has been used in many industrial fields. It is becoming important problems to estimate quantitatively not only the bonding strength of layer composites but also the thermophysical properties or characteristics of heat transfer.

Some transient methods, for example, the flash technique^{1),2)} and the stepwise heating technique³⁾ for determining the thermophysical properties of two- or three-layer composites have been already reported, but these methods can be only applied to layer composites without thermal contact resistance at the interlayer surfaces. In this study, a laser flash method of determining the thermal diffusivity of one layer of two-layer composites with the thermal contact resistance is proposed. The present method can be applied to the layer composites in which the thermal conductivity of the two layers and the thermal diffusivity of one layer are known. A general flash type thermal constant analyzer can be used, and the thermal diffusivity and the thermal contact resistance can be simultaneously determined by measuring the temperature history of the rear face in the same way of conventional

half-time method⁴⁾ for a homogeneous layer and carrying out a relatively easy numerical computation using an iteration method.

In this paper, the theoretical equations of the temperature increase of a two-layer composite due to an instantaneous heat source with uniform intensity is derived by Laplace transformation, and a method of determining the thermal diffusivity of one layer of the two-layer composite is described. 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, and the effectiveness of this method is confirmed.

2. Analysis of Temperature Distribution

The schematic diagram of the geometry of a two-layer composite is shown in Fig. 1. In the flash method, the front surface of the sample is subjected to a short radiant energy pulse and the resulting temperature history of the rear face is recorded. To solve the heat conduction equation with the appropriate boundary conditions and the initial conditions, the following assumptions are made:

- (1) one dimensional heat flow,
- (2) no heat loss from the sample surfaces,

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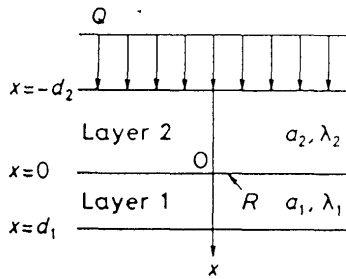


Fig. 1 Diagram of two-layer composite.

- (3) each layer is homogenous,
 (4) thermal contact resistance between layers is uniform,

and (5) heat pulse is uniformly absorbed on the front surface.

The heat conduction equation for each layer is mathematically described in the following way:

$$\left(\frac{\partial^2}{\partial x^2} - \frac{1}{a_k} \frac{\partial}{\partial t}\right) T_k(x, t) = 0, \quad k=1, 2. \quad (1)$$

The boundary conditions are

$$\frac{\partial T_1}{\partial x}(d_1, t) = 0, \quad (2)$$

$$\lambda_1 \frac{\partial T_1}{\partial x}(0, t) = \lambda_2 \frac{\partial T_2}{\partial x}(0, t) = \frac{1}{R} [T_1(0, t) - T_2(0, t)], \quad (3)$$

$$\lambda_2 \frac{\partial T_2}{\partial x}(-d_2, x) = -Q\delta(t), \quad (4)$$

and the initial conditions are

$$T_k(x, 0) = 0, \quad k=1, 2, \quad (5)$$

where T , a , λ , R and Q are the temperature increase, the thermal diffusivity, the thermal conductivity, the thermal contact 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, and $\delta(t)$ is the Dirac's delta function.

The equations for the Laplace transform are

$$\left(\frac{d^2}{dx^2} - q_k^2\right) \bar{T}_k(x, s) = 0, \quad k=1, 2, \quad (6)$$

with

$$\frac{d\bar{T}_1}{dx}(d_1, s) = 0, \quad (7)$$

$$\lambda_1 \frac{d\bar{T}_1}{dx}(0, s) = \lambda_2 \frac{d\bar{T}_2}{dx}(0, s) = \frac{1}{R} [\bar{T}_1(0, s) - \bar{T}_2(0, s)], \quad (8)$$

and

$$\lambda_2 \frac{d\bar{T}_2}{dx}(d_2, s) = -Q, \quad (9)$$

where

$$\bar{T}_k(x, s) = \int_0^\infty e^{-st} T_k(x, t) dt, \quad k=1, 2, \quad (10)$$

and

$$q_k^2 = \frac{s}{a_k}, \quad k=1, 2. \quad (11)$$

The solutions of the above equations are

$$\bar{T}_1(x, s) = \frac{2Q}{A} \cosh q_1(d-x), \quad (12)$$

$$\begin{aligned} \bar{T}_2(x, s) = & \frac{q_1 Q}{\lambda_2 q_2 A} [(\lambda_1 + \gamma \lambda_2) \cosh(d_1 - \gamma x) q_1 \\ & - (\lambda_1 - \gamma \lambda_2) \cosh(d_1 + \gamma x) q_1 \\ & + \gamma \lambda_1 \lambda_2 q_1 R [\sinh(d_1 - \gamma x) q_1 + \sinh(d_1 + \gamma x) q_1]], \end{aligned} \quad (13)$$

where

$$\gamma = \sqrt{\frac{a_1}{a_2}}, \quad (14)$$

and

$$\begin{aligned} A = & q_1 [(\lambda_1 + \gamma \lambda_2) \sinh(d_1 + \gamma d_2) q_1 \\ & + (\lambda_1 - \gamma \lambda_2) \sinh(d_1 - \gamma d_2) q_1 \\ & + \gamma \lambda_1 \lambda_2 q_1 R [\cosh(d_1 + \gamma d_2) q_1 - \cosh(d_1 - \gamma d_2) q_1]]. \end{aligned} \quad (15)$$

Using the Inversion Theorem for the Laplace transformation

$$T_k(x, t) = \frac{1}{2\pi i} \int_{\eta-i\infty}^{\eta+i\infty} e^{zt} \bar{T}_k(x, z) dz, \quad k=1, 2, \quad (16)$$

we have finally

$$\begin{aligned} T_1(x, t) = & \frac{a_1 Q}{\lambda_1 d_1 + \gamma^2 \lambda_2 d_2} \\ & + 4a_1 Q \sum_{n=1}^{\infty} \frac{\cos(d_1 - x) \alpha_n}{C_n} e^{-a_1 \alpha_n^2 t}, \end{aligned} \quad (17)$$

$$T_2(x, t) = \frac{a_1 Q}{\lambda_1 d_1 + \gamma^2 \lambda_2 d_2} + \frac{2a_1 Q}{\lambda_2 \gamma} \sum_{n=1}^{\infty} \frac{D_n(t)}{C_n} e^{-a_1 \alpha_n^2 t}, \quad (18)$$

where

$$\begin{aligned} C_n = & (\lambda_1 + \gamma \lambda_2)(d_1 + \gamma d_2) \cos(d_1 + \gamma d_2) \alpha_n \\ & + (\lambda_1 - \gamma \lambda_2)(d_1 - \gamma d_2) \cos(d_1 - \gamma d_2) \alpha_n \\ & - \gamma \lambda_1 \lambda_2 R [(d_1 + \gamma d_2) \sin(d_1 + \gamma d_2) \alpha_n \\ & - (d_1 - \gamma d_2) \sin(d_1 - \gamma d_2) \alpha_n] \alpha_n \\ & - [\cos(d_1 + \gamma d_2) \alpha_n - \cos(d_1 - \gamma d_2) \alpha_n], \end{aligned} \quad (19)$$

$$D_n(x) = (\lambda_1 + \gamma\lambda_2)\cos(d_1 - \gamma x)\alpha_n - (\lambda_1 - \gamma\lambda_2)\cos(d_1 + \gamma x)\alpha_n - \gamma\lambda_1\lambda_2\alpha_n R[\sin(d_1 - \gamma x)\alpha_n + \sin(d_1 + \gamma x)\alpha_n], \quad (20)$$

and the α_n is the n -th positive root of

$$(\lambda_1 + \gamma\lambda_2)\sin(d_1 + \gamma d_2)\alpha + (\lambda_1 - \gamma\lambda_2)\sin(d_1 - \gamma d_2)\alpha + \alpha\gamma\lambda_1\lambda_2 R[\cos(d_1 + \gamma d_2)\alpha - \cos(d_1 - \gamma d_2)\alpha] = 0. \quad (21)$$

T_k converges to the first term of Eqs. (17) or (18) when $t \rightarrow \infty$, because the terms expressed by the infinite series converge to zero as the time t increases. The first term is the maximum temperature increase of the sample, which is independent of the thermal contact resistance at the interlayer surfaces. Therefore, the temperature increase on the rear face, $x = d_1$, can be normalized as Eq. (22), using the first term.

$$\Theta = 1 + 4(\lambda^* d^* + \gamma^2) \sum_{n=1}^{\infty} \frac{e^{-\beta_n^2 \gamma^2 t^*}}{\left[\begin{aligned} &(\lambda^* + \gamma)(d^* + \gamma) \cos \beta_n (d^* + \gamma) \\ &+ (\lambda^* - \gamma)(d^* - \gamma) \cos \beta_n (d^* - \gamma) \\ &- \gamma \lambda^* R^* \{ \beta_n [(d^* + \gamma) \sin \beta_n (d^* + \gamma) \\ &- (d^* - \gamma) \sin \beta_n (d^* - \gamma)] \\ &- [\cos \beta_n (d^* + \gamma) - \cos \beta_n (d^* - \gamma)] \} \end{aligned} \right]}, \quad (22)$$

where

$$\lambda^* = \frac{\lambda_1}{\lambda_2}, \quad d^* = \frac{d_1}{d_2}, \quad R^* = \frac{\lambda_2 R}{d_2}, \quad t^* = \frac{a_2 t}{d_2^2}, \quad \beta_n = \alpha_n d_2, \quad (23)$$

and

$$\Theta = \left(1 + \frac{\lambda_1 a_2 d_1}{\lambda_2 a_1 d_2}\right) \frac{\lambda_2 d_2}{a_2 Q} T_1(d_1, t). \quad (24)$$

3. Theory of Measurement

We suppose that, for example, the thermal diffusivity of ceramics layer of a two-layer specimen composed of a metal and a ceramics layer is unknown. If the laser beam is irradiated on the surface of the ceramics layer in the laser flash method, the beam penetrates into the ceramics layer⁵⁾, therefore, the boundary condition differs from Eq. (4). It is appropriate that the laser beam is irradiated on the metal surface and the temperature increase on the surface of the ceramics layer is measured. Therefore, postulating that the thermal diffusivity of the layer 1 in Fig. 1 is unknown, we consider the measuring method of a_1 and the thermal contact resistance R in this section.

Figure 2 shows some examples of normalized temperature versus dimensionless time curves on the rear face obtained by calculating Eq. (22). Θ is a monotone increasing function and converges to one when $t^* \rightarrow \infty$, in-

dependently of the thermophysical properties and the thermal contact resistance of the two-layer composite. Because γ and R^* are unknowns and t^* is a variable in Eq. (22), we can represent Θ in the following form

$$\Theta = \Theta(\gamma, R^*; t^*). \quad (25)$$

We introduce a new parameter t_α^* defined by the following equation

$$\Theta(\gamma, R^*; t_\alpha^*) = \alpha, \quad (26)$$

where $0 < \alpha < 1$.

Let the temperature versus time curve of $\gamma = 0.1$ and $R^* = 10$, that is, the curve P in Fig. 2 be obtained by temperature measurement, then $t_{0.3}^*$ and $t_{0.7}^*$, for example, can be read as 7.72 and 16.74 from the figure when α is set at 0.3 and 0.7, respectively. When t_α^* is known and γ and R^* are variables, the combinations of γ and R^* which satisfy Eq. (26) are expressed as the γ - R^* curves, as shown in Fig. 3, and these curves are crossed at a point P, which shows the values of γ and R^* to be obtained. Therefore, if we express γ which satisfies Eq. (26) for any

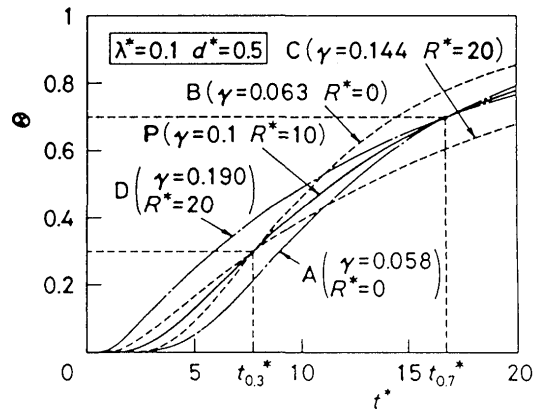


Fig. 2 Normalized temperature rise versus dimensionless time curves on the rear face.

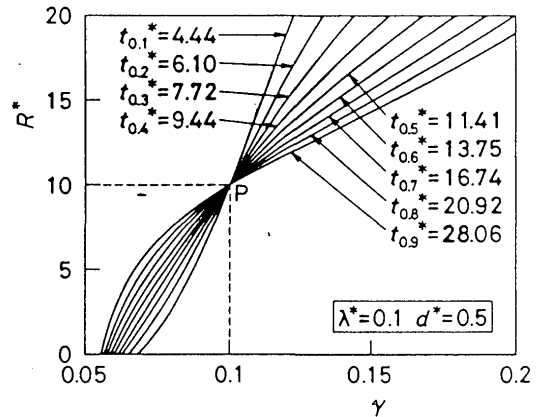


Fig. 3 γ - R^* curves.

R^* like as $\gamma(R^*; t_{\alpha}^*)$, both γ and R^* can be determined by solving the equation

$$\gamma(R^*; t_{\alpha_1}^*) - \gamma(R^*; t_{\alpha_2}^*) = 0 \quad (27)$$

which holds for two α -values, α_1 and α_2 . This solution can be obtained easily through the numerical calculation. In this study, the method of bisection algorithm was adopted, and the numerical calculations were carried out on a minicomputer, HITAC E-800. The iteration was stopped when R^* at the $(n+1)$ th step satisfied the following condition

$$\left| \frac{R^{*(n+1)} - R^{*(n)}}{R^{*(n)}} \right| < \varepsilon, \quad (28)$$

where ε is a positive value and the superscript (n) denotes the value which is obtained at the n -th iteration. **Table 1** shows the iteration times and computational time for convergence to the solution for some combinations of the initial values of R^* and the positive values ε . For many numerical examples, the solutions were also obtained in relatively short time as shown in Table 1.

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 data acquisition system. The pulse width of the laser is about 0.7ms. The temperature detector used was a radiation thermometer (InSb sensor) and the temperature history on the rear face of the sample was recorded by a transient memory and a pen recorder.

We used three kinds of materials, that is, carbon steel for machine structural use, S45C, and tool steel, SK5, and stainless steel, SUS 304 which are equivalent to AISI 1045, ASTM W1-8 and AISI 304, respectively. The thermophysical properties of these materials are influenced by the contents. Therefore, the thermal diffusivity and

Table 1 Iteration times n and computational time used for convergence to the solution.

| $R^*(1)$ | 0.5×10^{-2} | | 0.5×10^{-3} | | 0.5×10^{-4} | |
|----------|----------------------|-----------|----------------------|-----------|----------------------|-----------|
| | n | Time s | n | Time s | n | Time s |
| 0.01 | 18 | 46.8 | 22 | 55.1 | 25 | 61.9 |
| 0.1 | 15 | 38.5 | 19 | 46.9 | 22 | 53.9 |
| 1 | 13 | 32.5 | 16 | 38.6 | 19 | 45.2 |
| 10 | 10 | 23.6 | 13 | 30.7 | 17 | 39.8 |
| 100 | 12 | 26.6 | 16 | 35.1 | 19 | 41.8 |
| 1000 | 16 | 34.1 | 19 | 40.4 | 22 | 46.9 |

conductivity of these materials were measured by the same apparatus using the conventional half-time method of the laser flash technique for a homogeneous layer, which are shown in **Table 2**. These properties will be used as the known values of λ_1 , λ_2 and a_2 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 of diameter 10mm and thickness from 0.7 to 0.8mm. 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 welding force, welding current and weld time were set at 3.04×10^5 Pa, 13.5kA and 133ms, 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. Results

An example of the recorded temperature history on the rear face for a spot-welded composite of SUS 304 stainless steel and S45C carbon steel is shown in **Fig. 4**, where the laser beam was irradiated on the surface of the SUS 304 layer. The drastic increase of temperature at the laser irradiating time may be caused by the direct input of a part of laser beam into the radiation thermometer. In this experiment, $t_{0.3}$ and $t_{0.7}$ were read from the temperature versus time curves, as shown in **Fig. 4**, and the thermal diffusivity a_1 of the layer 1 and the thermal contact resistance R at the interlayer surface were measured by the present method using the thermophysical properties shown in **Table 2**.

Table 3 shows the $t_{0.3}$ and $t_{0.7}$ measured, and a_1 and R obtained by the present measuring method. The relative errors of obtained in **Table 3** to the thermal diffusivity

Table 2 Thermal diffusivity and conductivity of materials used.

| Material | a m ² /s | λ W/(m·K) |
|----------|-----------------------|-------------------|
| S45C | 1.10×10^{-5} | 38.2 |
| SK5 | 1.06×10^{-5} | 38.9 |
| SUS304 | 3.55×10^{-6} | 13.1 |

shown in Table 2 are within about $\pm 7\%$ and $\pm 30\%$ for the spot-welded and silicon-linked specimens, respectively, and the latter is about four times as large as the former. It is interpreted for this result that the silicon-linked specimen is not a strict two-layer composite but a three-layer one, because there is a very thin film of silicon grease at the interface of the two layers. The thermal contact resistance is about from 10^{-6} to $10^{-5} \text{m}^2\text{K/W}$ for the specimen by spot welding, which differs a little with specimens. On the other hand, it is almost $1 \times 10^{-4} \text{m}^2\text{K/W}$ for all specimens by silicon linking.

If all thermophysical properties of the two-layer composites are known, the thermal contact resistance at the interlayer surface can be also determined by the laser flash method⁶⁾. The method is summarized as follows: Because R^* is an unknown and t^* is a variable in Eq. (22), we can represent θ in the following form

$$\theta = \theta(R^*; t^*), \tag{29}$$

and R^* can be easily determined by solving

$$\theta(R^*; t_{\alpha}^*) = \alpha, \tag{30}$$

through the numerical computation; such as the method of bisection algorithm or regula falsi.

The thermal contact resistance obtained by the above measuring method, where α was set at 0.5, are shown in Table 4. The relative errors of R^* obtained in Table 3 to the results shown in Table 4 are about $\pm 4\%$ for the spot-welded composite of SUS 304 and the silicon-linked composite of SK5 tool steel, and 7% for the silicon-linked composite of S45C carbon steel and SUS 304 stainless steel. The maximum relative error of R^* is about -28% for the silicon-linked composite of SUS 304 stainless steel. For other specimens, the relative error is about from 10 to 20%, therefore, it is estimated that the thermal contact resistance was also obtained with almost same accuracy for the thermal diffusivity.

6. Conclusion

A laser flash of determining the thermal diffusivity of one layer of two-layer composites with the thermal contact resistance at the interlayer surface is proposed. In the present method, the time required for the temperature to

Table 3 Measured results of thermal diffusivity and thermal contact resistance of some two-layer composites.

| Joint method | Layer | | d_1 mm | d_2 mm | $t_{0.3}$ ms | $t_{0.7}$ ms | α m^2/s | R $\text{m}^2\text{K/W}$ |
|-----------------|--------|--------|-------------|-------------|-----------------|-----------------------|-----------------------------------|-------------------------------|
| | 1 | 2 | | | | | | |
| Spot welding | S45C | SUS304 | 0.767 | 0.768 | 44.5 | 87.5 | 1.12×10^{-5} | 2.81×10^{-6} |
| | SK5 | SUS304 | 0.810 | 0.767 | 53.3 | 108.5 | 1.13×10^{-5} | 1.39×10^{-5} |
| | SUS304 | SUS304 | 0.777 | 0.777 | 86.5 | 175.8 | 3.60×10^{-5} | 2.79×10^{-5} |
| Silicon linking | S45C | S45C | 0.781 | 0.773 | 79 | 220 | 9.37×10^{-6} | 1.00×10^{-4} |
| | SK5 | S45C | 0.783 | 0.773 | 93 | 275 | 1.37×10^{-5} | 1.63×10^{-4} |
| | SK5 | SK5 | 0.783 | 0.781 | 101 | 290 | 8.46×10^{-6} | 1.26×10^{-4} |
| | S45C | SUS304 | 0.773 | 0.780 | 100 | 250 | 1.00×10^{-5} | 9.34×10^{-5} |
| | SK5 | SUS304 | 0.783 | 0.778 | 130 | 342 | 7.54×10^{-4} | 1.21×10^{-4} |
| SUS304 | SUS304 | 0.778 | 0.780 | 144 | 332 | 2.54×10^{-6} | 8.96×10^{-5} | |

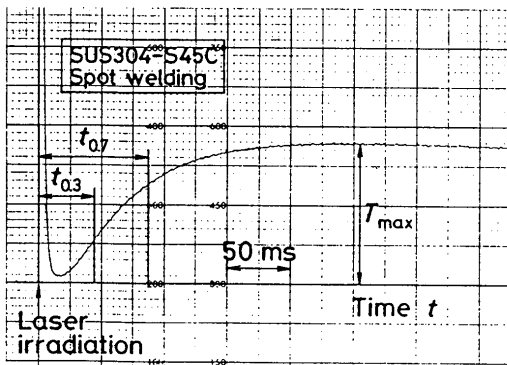


Fig. 4 An example of the recorded temperature history on the rear face.

Table 4 Measured results of thermal contact resistance of the same specimens shown in Table 3, which were obtained by another measuring method.

| Joint method | Layer | | $t_{0.5}$ ms | R $\text{m}^2\text{K/W}$ |
|-----------------|--------|--------|-----------------------|-------------------------------|
| | 1 | 2 | | |
| Spot welding | S45C | SUS304 | 62.0 | 2.32×10^{-6} |
| | SK5 | SUS304 | 75.5 | 1.19×10^{-5} |
| | SUS304 | SUS304 | 122.5 | 2.69×10^{-5} |
| Silicon linking | S45C | S45C | 138 | 1.14×10^{-4} |
| | SK5 | S45C | 162 | 1.35×10^{-4} |
| | SK5 | SK5 | 180 | 1.31×10^{-4} |
| | S45C | SUS304 | 160 | 1.00×10^{-4} |
| | SK5 | SUS304 | 219 | 1.55×10^{-4} |
| SUS304 | SUS304 | 221 | 1.25×10^{-4} | |

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. The thermal contact resistance can be obtained easily as well as the thermal diffusivity. 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, and the effectiveness of this method is confirmed. The thermal contact resistance is also measured in addition by the another method already proposed by authors, and these obtained results are in fairly good agreement each other.

The present method will be useful for quantitative estimation of the thermophysical properties or characteristics of heat transfer of the two-layer composite specimens composed of a ceramics layer and a metal, and so on.

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