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Citation	Transactions of JWRI. 1989, 18(1), p. 59-64
Version Type	VoR
URL	https://doi.org/10.18910/7970
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Determination 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 of 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 theiry- 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 authors[1] 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 method[2]. 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

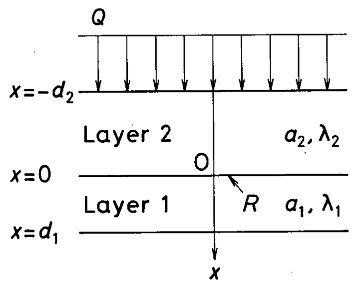

Fig. 1 Diagram of two-layer composite.

† Received on May 8, 1989

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Transactions of JWRI is published by Welding Research Institute of Osaka University, Ibaraki, Osaka 567, Japan

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 dimentional 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 nomalized temperature of the rear surface is represented as Eq. (1)[1].

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

where

$$\left.\begin{array}{l} \Theta = (1 + \dfrac{\lambda_1 \alpha_2 d_1}{\lambda_2 \alpha_1 d_2}) \dfrac{\lambda_2 d_2}{a_2 Q} T_1(d_1, t), \\ \gamma = \sqrt{\dfrac{a_1}{a_2}}, \lambda^* = \dfrac{\lambda_1}{\lambda_2}, d^* = \dfrac{d_1}{d_2}, R^* = \dfrac{\lambda_2 R}{\lambda_2}, \\ t^* = \dfrac{a_2 t}{d_2^2}, \end{array}\right\} \quad (2)$$

and T, a, λ, 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 β_n is the n-th positive root of

$$(\lambda^* + \lambda)\sin\beta(d^*+\gamma) + (\lambda^*-\gamma)\sin\beta(d^*-\gamma) + \beta\gamma\lambda^* R^* [\cos\beta(d^*+\gamma) - \cos\beta(d^*-\gamma)] = 0. \quad (3)$$

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

$$\Theta = \Theta(\gamma, R^*; t^*). \quad (4)$$

Θ is a monotone increasing function of t^* and converges to one when $t^* \to \infty$, 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

$$\theta(\tau, R^*; t_\alpha{}^*) = \alpha, \quad (5)$$

where $0 < \alpha < 1$.

When $t_\alpha{}^*$ is known and γ and R^* are variables, the combination of γ and R^* which satisfies Eq. (5) is expressed as a curved line in the γ-R^* plane[1] 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 γ and R^* to be obtained. Therefore, if we express γ which satisfies Eq. (5) for any R^* like as $\gamma(R^*; t_\alpha{}^*)$, both γ and R^* can be determined by solving the equation

$$\gamma(R^*; t_{\alpha_1}{}^*) - \gamma(R^*; t_{\alpha_2}{}^*) = 0, \quad (6)$$

which holds for the 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 addopted.

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 ϵ for the true values, d_i, λ_j, a_2 and $t_{\alpha_k}(i, j, k = 1, 2)$, we can express these parameters are

$$\left.\begin{array}{ll} d_i' = (1+\epsilon_{d_i}) d_i, & i = 1, 2 \\ \lambda_j' = (1+\epsilon_{\lambda_j})\lambda_j, & j = 1, 2 \\ a_2' = (1+\epsilon_{a_2}) a_2, & \\ t_{\alpha_k}' = (1+\epsilon_{t_{\alpha_k}}) t_{\alpha_k}, & k = 1, 2. \end{array}\right\} \quad (7)$$

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

$$\left.\begin{array}{l} d^{*\prime} = \dfrac{1+\epsilon_{d_1}}{1+\epsilon_{d_2}} d^*, \\ \lambda^{*\prime} = \dfrac{1+\epsilon_{\lambda_1}}{1+\epsilon_{\lambda_2}} \lambda^*, \\ t_{\alpha_k}{}^{*\prime} = \dfrac{(1+\epsilon_{\alpha_2})(1+\epsilon_{t_{\alpha_k}})}{(1+\epsilon_{d_2})^2} t_{\alpha_k}{}^*, \ k = 1, 2. \end{array}\right\} \quad (8)$$

The values of γ and R^* obtained through the present measuring method using the above values have relative errors ϵ_γ and ϵ_{R*}, and they are written as follows:

$$\left.\begin{aligned} \gamma' &= (1+\epsilon_\gamma)\gamma, \\ R^{*\prime} &= (1+\epsilon_{R*})R^*, \end{aligned}\right\} \tag{9}$$

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

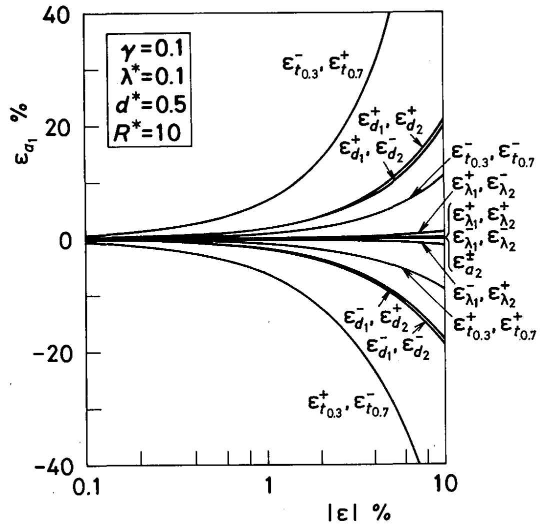
$$\left.\begin{aligned} a_1{}' &= (1+\epsilon_\gamma)^2(1+\epsilon_{\alpha_2})a_1 \\ R' &= \frac{(1+a_{d_2})(1+\epsilon_{R*})}{1+\epsilon_{\lambda_2}}R \end{aligned}\right\} \tag{10}$$

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

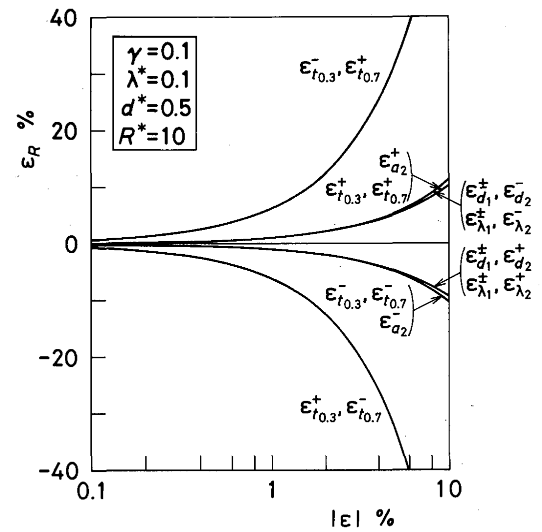
$$\epsilon_{\alpha_1} = (1+\epsilon\gamma)^2(1+\epsilon_{\alpha_2})-1, \tag{11}$$

$$\epsilon_R = \frac{(1+\epsilon_{d_2})(1+\epsilon_{R*})}{1+\epsilon_{\lambda_2}} - 1. \tag{12}$$

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 α_1 and α_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 ϵ_{a_1} and ϵ_R is shown in this figure. Either ϵ^+ or ϵ^- 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 ϵ_{a_1} and ϵ_R increase as the relative error of each parameter increases. Especially, when $\epsilon_{t_{0.3}}$ and $\epsilon_{t_{0.7}}$ have different sign, the relative errors of the estimated values become largest, and the errors of thickness are followed. The errors of thermal conductivities λ_i (i = 1, 2) and thermal diffusivity a_2 do not affect the final accuracy so much.



(a) Relative error of thermal diffusivity, a_1.



(b) Relative error of thermal resistance, R.

Fig. 2 Influence of each relative error of d_i, λ_i, $t_{0.3}$ and $t_{0.7}$ on accuracy of a_1 and R obtained.

Table 1 Thermal diffusivity and conductivity of materials used.

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

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 cacuum 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 thermophysical properties of these materials are influenced by the contents. Therefore, the thermal diffusivity a and the heat capacity ρc 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[3], respectively, then the thermal conductivity was calculated using the relation of $\lambda = (\rho c)a$. The measured results are shown in **Table 1**.

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 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 3.04×10^5 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, α_1 and α_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 specines 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 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 ◇ and ◆, 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 ones 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 ◇ and ◆, 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 indecate that the values are concentrated on the mean value relatively well. The five points of the first setting, which are shown by the symbol ○, 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,

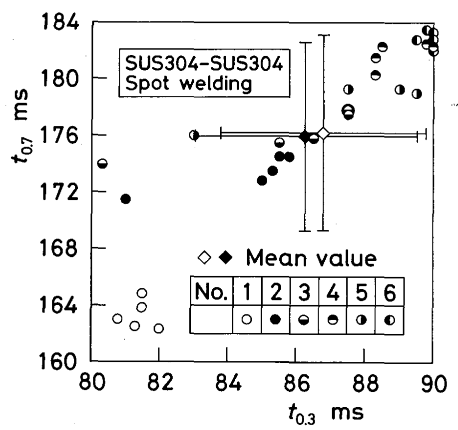


Fig. 3 Scatter in measured values of $t_{0.3}$ and $t_{0.7}$.

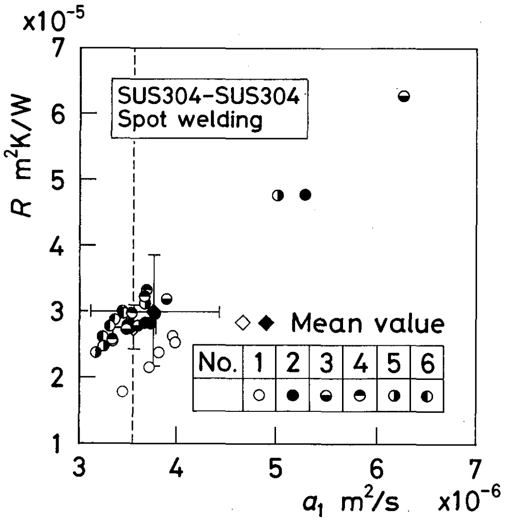
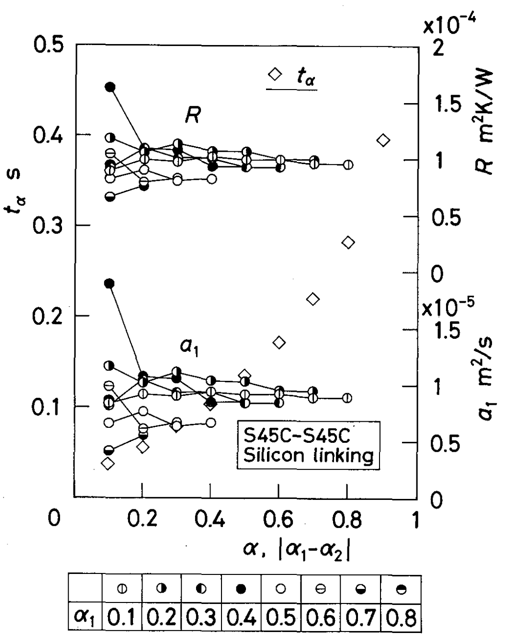
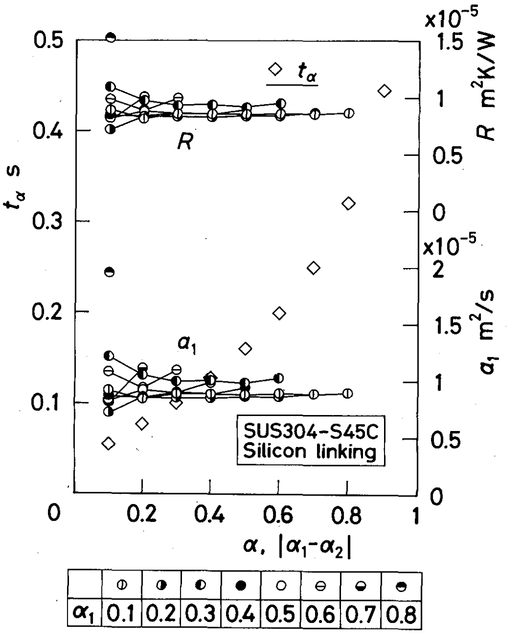


Fig. 4 Measured results of a_1 and R with $t_{0.3}$ and $t_{0.7}$ shown in Fig. 3.



(a) A two-layer composite of S45C carbon steel by silicon-linking.



(b) A two-layer composite of SUS304 stainless steel and S45C carbon steel by silicon-linking.

Fig. 5 Measured results of a_1 and R with various combinations of $t_{0.3}$ and $t_{0.7}$.

the relative errors of a_1 and R are smaller. This conclusion is in good agreement with the results that, as shown in Fig. 2, the measured errors are not so large when the errors of the times $t_{0.3}$ and $t_{0.7}$ have same sign, on the other hand, they become largest when the errors of them have different sign.

In the above measurement, the times $t_{0.3}$ and $t_{0.7}$ were read from the temperature versus time curves. The present method can be also applied for any α_1 and α_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_{α_1} and t_{α_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 corespond to t_α and the absolute of the difference between α_2 and α_1. The circles in the figures mean the values of α_1, which are shown in the tables under the figures. As seen from Fig. 5, the amount of scatter in measured values of α_1 and R is larger as the difference between α_1 and α_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[1)] 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 α_1 and α_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_{α_1} and t_{α_2} read from the temperature versus time curve of the rear surface. If the amount of scatter of the time t_{α_1} and t_{α_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 α_1 and α_2 is smaller. It is proper to select the values of about 0.3 and 0.7 for α_1 and α_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.

Acknowledgement

The authors wish to express their gratitude to Matsushita Industrial Equipment Co., Ltd. for its useful advice and support with offer of some experimental apparatus on making specimens. The authors also would like to thank Messrs. T. Sugimachi and M. Tachibana in their laboratory for their considerable assistance.

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