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Measurement of Thermal Diffusivity of Thermal Control Coatings by the Flash Method Using Two-Layer Composite Sample[1]

G. H. He,[2–4] J. D. Guo,[2, 3] Y. Y. Zhang,[2] B. Q. Wang,[2] and B. L. Zhou[2, 3]

The thermal diffusivity of brittle coatings cannot be measured by the flash method directly because of the difficulty of preparing free-standing samples. Adopting the flash method using a two-layer composite sample, it is possible to measure thermal diffusivity if the radiant pulse is well defined and good thermal contact on the interface of the composite sample can be ensured. Using an equilateral trapezoidal pulse of an Nd-glass laser measuring the dimensionless temperature history of the rear face of the sample, we determined the thermal diffusivity of thermal control coatings in the temperature range of 80 to 200°C. The results for different thicknesses of substrate showed that the thermal contact resistance of the interface can be neglected.

KEY WORDS: flash method; thermal contact resistance; thermal control coatings; thermal diffusivity; two-layer composite sample.

1. INTRODUCTION

The thermal diffusivity and thermal conductivity of thermal control coatings play an important role in satellite thermal control design. For these thin coatings, it is difficult to measure the thermal conductivity by the steady-state method. The flash method [1] for measuring the thermal diffusivity of thin samples is recommended. However, the difficulty of preparing free-standing samples of brittle coatings becomes a significant problem.

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[2] Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110015, People's Republic of China.

[3] International Center for Materials Physics, Chinese Academy of Sciences, Shenyang 110015, People's Republic of China.

[4] To whom correspondence should be addressed.

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Thus, it is necessary to adopt the flash method using two-layer composite samples.

The temperature rise curve for the rear face of the composite sample is different for different heat-pulse functions. For our experiments, we use the heat pulse of an Nd-glass laser that takes the form similar to an equilateral trapezoid, the solution of which has been deduced previously [2]. In order to diminish the effects of thermal contact resistance in the composite samples, we used several specimens that have substrate layers with different thicknesses. If the thermal diffusivities of coatings for samples with different thicknesses of substrate layers have very similar values, we consider that the thermal contact resistance can be neglected. Furthermore, the substrate of the composite sample is selected according to the optimum combination of thermal properties and thicknesses for the two layers.

2. PRINCIPLE AND CALCULATION

2.1. Measurement Principle

For a two-layer composite sample (Fig. 1) irradiated by an equilateral trapezoidal laser pulse (Fig. 2), the temperature rise function of the rear face can be expressed as [2]

$$V(L_2, t) = 1 + \frac{2U_2^2}{b(1-b)\,\tau^2} \sum_{k=1}^{\infty} \frac{\exp(-\beta_k^2 t/U_2)}{\beta_k} \times \frac{1 + \exp(\tau\beta_k^2/U_2) - \exp(b\tau\beta_k^2/U_2) - \exp[(1-b)\,\tau\beta_k^2/U_2]}{\cos(\beta_k X)\cos\beta_k - \Omega(X)\sin(\beta_k X)\sin\beta_k} \quad (1)$$

where $U_i = L_i^2/\alpha_i$, $X = (U_1/U_2)^{1/2}$, $H = C_1 L_1 \rho_1 / C_2 L_2 \rho_2$, and $\Omega(X) = (X + HX^{-1})/(H+1)$. Here C_i, L_i, and ρ_i are the specific heat, thickness,

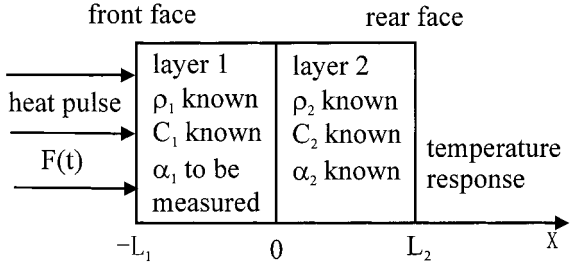

Fig. 1. Model of two-layer composite sample.

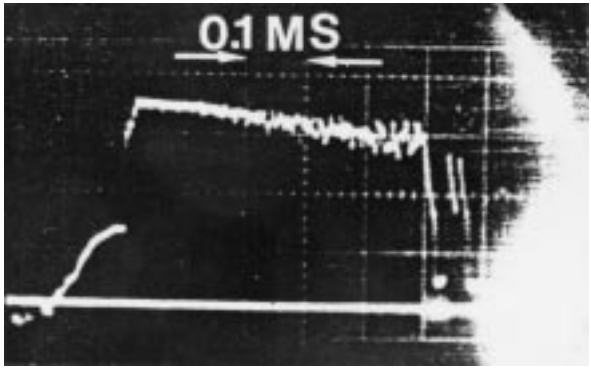

Fig. 2. Heat pulse shape.

and density of the two layers, respectively, τ is the pulse duration of the Nd-glass laser, $b\tau$ is the rise and fall duration of the trapezoid, and β_k is the kth positive root of the following equation:

$$H \sin(\beta_k X) \cos \beta_k + X \cos(\beta_k X) \sin \beta_k = 0 \tag{2}$$

2.2. Calculation of Thermal Diffusivity

When $V(L_2, t) = 0.5$, where $t\ (= t_{1/2})$ is the time of half maximum temperature of the rear face of a composite sample, we can use an iterative method to calculate the thermal diffusivity of coatings α_1. If we can achieve a calculation precision of 0.0001 for $V(L_2, t)$ and of 0.01 for β_k, we can consider the value α_1 to be the desired thermal diffusivity.

3. EXPERIMENTS AND RESULTS

3.1. Experimental System

The main apparatus for the thermal diffusivity measurement has been described elsewhere [3]. For the experiments at 80 to 200°C, an InSb detector has been used instead of the PbS detector. The experimental system is shown in Fig. 3. The nonlinear response of the InSb detector has been discussed by Hoefler and Taylor [5]. They pointed out that this nonlinear behavior can shift the $t_{1/2}$ to larger values and cause lower thermal diffusivities, especially at room temperature and for large temperature excursions. In the present experiments, the test temperature is higher than

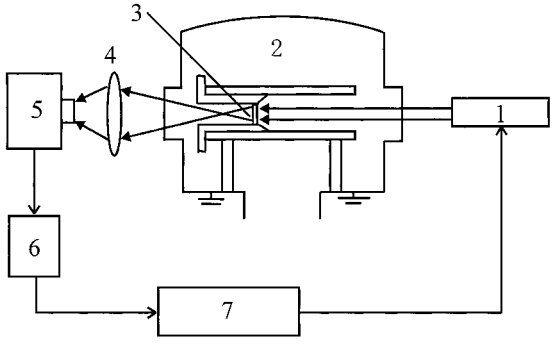

Fig. 3. Experimental system. (1) Nd-glass laser, (2) vacuum furnace, (3) sample, (4) CaF_2 lens, (5) InSb detector, (6) amplifier, (7) computer.

85°C and the temperature excursion is only 2 to 3°C. Thus, it is reasonable to disregard the effects of the nonlinearity of the InSb detector.

3.2. Sample Preparation and Experiments

We chose an Al–Mg alloy (Al: 94.66 wt%, Mg: 4.74 wt%, Mn: 0.6 wt%) as the substrate (layer 2), and prepared two samples (A and B) which have the same diameter of 14.82 mm and thicknesses of 2.88 and 2.42 mm, respectively. After the mass and the density of the samples were measured, the coating (i.e., layer 1) was sprayed on one face of samples A and B. Then the composite samples were treated at appropriate temperatures. The final thicknesses of composite samples A and B were 3.075 and 2.615 mm, respectively. We weighed them again and obtained the density of the coatings. The specific heat of the substrate can be calculated by the Kopp–Neumann law [4] according to the composition of the Al–Mg alloy. The specific heat of the coatings was measured by a DSC apparatus.

3.3. Results

The specific heat of substrate and coatings is shown in Fig. 4. The thermal diffusivity of Al–Mg alloy, using another sample with a thickness of 3.92 mm, was measured, and the results are shown in Fig. 5. The temperature rise curve of the composite samples A and B was measured, and the $t_{1/2}$ values were recorded. Then the thermal diffusivities of the coatings were calculated using Eqs. (1) and (2) with $\tau = 0.0007$ s and $b = 0.1$. The results are shown in Table I and Fig. 6.

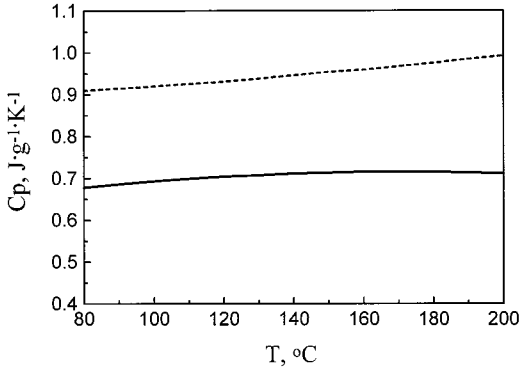

Fig. 4. Specific heat of two layers. Solid line: coating; dashed line: Al–Mg alloy.

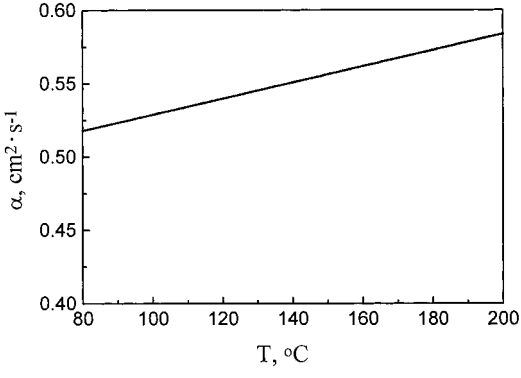

Fig. 5. Thermal diffusivity of Al–Mg alloy.

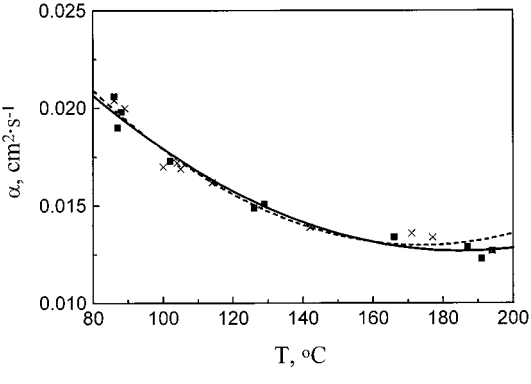

Fig. 6 Thermal diffusivity of coatings for composite samples A (solid line) and B (dashed line).

Table I. Thermal Diffusivity of Coatings

	Temperature (°C)								
Sample A[a, b]	86	87	102	126	129	166	187	191	194
$t_{1/2}$ (ms)	33.4	34.0	34.7	35.7	35.4	36.0	36.0	36.6	36.1
α_t ($cm^2 \cdot s^{-1}$)	0.0206	0.0190	0.0173	0.0149	0.0151	0.0134	0.0129	0.0123	0.0127
α_c ($cm^2 \cdot s^{-1}$)	0.0198	0.0197	0.0177	0.0152	0.0150	0.0130	0.0127	0.0127	0.0127
δ (%)	+4.04	−3.55	−2.26	−1.97	+0.67	+3.07	+1.57	−3.15	0
X	0.340	0.355	0.375	0.408	0.404	0.441	0.452	0.464	0.460
	Temperature (°C)								
Sample B[b, c]	86	89	104	105	114	142	171	177	194
$t_{1/2}$ (ms)	26.4	26.5	27.6	27.8	28.0	29.2	28.9	29.0	29.4
α_t ($cm^2 \cdot s^{-1}$)	0.0204	0.0200	0.0172	0.0169	0.0162	0.0139	0.0136	0.0134	0.0127
α_c ($cm^2 \cdot s^{-1}$)	0.0199	0.0194	0.0174	0.0172	0.0162	0.0138	0.0130	0.0130	0.0133
δ (%)	+2.51	+3.1	−1.15	−1.74	0	+0.72	+4.62	+3.08	−4.52
X	0.406	0.430	0.448	0.450	0.460	0.510	0.520	0.524	0.546

[a] Sample A: $L_1 = 0.0195$ cm, $L_2 = 0.288$ cm, $\rho_1 = 2.32\ g \cdot cm^{-3}$, $\rho_2 = 2.65\ g \cdot cm^{-3}$.
[b] Relative deviation of α_t from α_c, $\delta = (\alpha_t - \alpha_c)/\alpha_c$, where α_t is the measured value and α_c is the smooth value.
[c] Sample B: $L_1 = 0.0195$ cm, $L_2 = 0.242$ cm, $\rho_1 = 2.27\ g \cdot cm^{-3}$, $\rho_2 = 2.65\ g \cdot cm^{-3}$.

4. DISCUSSION

For the flash method using a two-layer composite sample, it is important to obtain the appropriate combination for the two layers. In general, the thermal diffusion time of heat flow through each of the two layers should be as similar as possible, i.e., $X \approx 1$ in Eqs. (1) and (2). According to satellite thermal control design, the thicknesses of the coatings should be 0.2 mm or so, and the substrate materials should be aluminum alloys. Thus, the selection of the thicknesses of the two layers may be limited. We chose the thickness of layer 2 to be 2.88 and 2.42 mm and obtained the results shown in Table I and Fig. 6. It can be seen from Table I that the smaller the thickness of layer 2, the larger the value of X. If we chose the thickness of layer 2 smaller than 2.42 mm, the value of X can be varied to near 1, but the $t_{1/2}$ might be reduced and the measurement error might be increased. Fortunately, although the values of X are between ~ 0.35 and ~ 0.55 for composite samples A and B, the deviation of the thermal diffusivity for coatings from smoothed data is less than $\pm 5\%$.

In the thermal control design, it is necessary to get good adhesion and good thermal contact between substrate and coatings. In order to check the effects of thermal contact resistance on the measurements, we prepared two samples with different thicknesses of substrate layers as shown above for sample A and sample B. If no thermal contact resistance exists, the thermal diffusivities of the coatings should have very similar values for the two samples. One can assume that the thermal contact resistance results in a decrease in the thermal diffusivity or a slight increase in $t_{1/2}$ relative to that of normal conditions. We used the same $t_{1/2}$ increments, for example, from 2 to 20 ms, to calculate the thermal diffusivity of coatings for sample A (86°C) and sample B (86°C), and obtained the results as shown in Table II.

It can be seen from Table II that the thermal diffusivity of the coatings will decrease substantially with the increase of $t_{1/2}$ if thermal contact resistance exists. Because the thermal contact resistance might not be known, the thermal diffusivity of α_1 for sample A might differ (for example, by more than $\pm 5\%$) from that for sample B if thermal contact resistance exists in the two samples or only in one of them. For example, if $\alpha_1 = 0.01397\ \text{cm}^2 \cdot \text{s}^{-1}$ for sample A and No. 3, and $\alpha_1 = 0.01646\ \text{cm}^2 \cdot \text{s}^{-1}$ for sample B and No. 2 (see Table II), then the difference in α_1 is more than 15%. In fact, the difference of thermal diffusivity data between the composite samples A and B is less than $\pm 5\%$ (see Table II and Fig. 6). In the experiments, the data must be discarded if the thermal diffusivity of the coatings has values that differ by more than $\pm 10\%$ between two samples; then new samples must be prepared. Thus, the effects of thermal contact

Table II. Variation of Thermal Diffusivity in $t_{1/2}$ Increments[a]

No.	$t_{1/2}$ Increment	α_1 ($\text{cm}^2 \cdot \text{s}^{-1}$)	
		Sample A (86°C)	Sample B (86°C)
1	0	0.02058	0.02043
2	2	0.01664	0.01646
3	4	0.01397	0.01365
4	6	0.01198	0.01162
5	8	0.01049	0.01012
6	10	0.009267	0.008915
7	15	0.007170	0.006856
8	20	0.005806	0.005548

[a] $t_{1/2} = 33.4$ ms, measured value for sample A; $t_{1/2} = 26.4$ ms, measured value for sample B.

resistance of the interface can be neglected if one prepares the composite samples carefully with different thicknesses of substrate. This would ensure reliability of thermal diffusivity measurements on the coatings.

5. CONCLUSION

The thermal diffusivity of brittle coatings with a thickness of about 0.2 mm can be measured by the flash method using two-layer composite samples and a radiation pulse of equilateral trapezoidal form. By using different thicknesses of substrate layers and preparing the composite samples carefully, the effects of thermal contact resistance of the interface can be neglected and reliable measurement of the thermal diffusivity of thermal control coatings can be made.

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