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Evaluation of the Effective Sample Temperature in Thermal Diffusivity Measurements Using the Laser Flash Method

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In the measurement of thermal diffusivity by the laser flash method, a temperature rise occurs in the sample as a pulsed laser hits on the sample surface. Due to the temperature dependence of thermal diffusivity of the sample, the thermal diffusivity corresponds to a temperature that is larger by T_{eff} than the temperature before laser irradiation is applied. This effective temperature rise, T_{eff}, has been investigated by using a numerical simulation. The results indicate that the effective temperature rise is almost equal to a maximum temperature rise, T_{M}, of the back surface of the sample in cases where both linear and nonlinear temperature variations of thermal diffusivity are considered.

KEY WORDS: glassy carbon; laser flash method; temperature dependence; thermal diffusivity; pulse width.

1. INTRODUCTION

The flash method proposed by Parker et al. [1] is one of the most popular techniques for measuring the thermal diffusivity of solids. In this technique, one side of a disk sample is irradiated by a pulsed laser or a xenon flash

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lamp, and the temperature rise of its back surface is measured as a function of time from which the thermal diffusivity value can be determined.

Front face temperature rises can be quite large for black insulating materials. All of this energy is originally concentrated in a very thin section and diffuses to the rest of the sample, raising the overall temperature several degrees. The temperature rise of the sample surface is up to 100 degrees. The temperature dependence of the thermal diffusivity and the effect of the temperature rise in the sample should be taken into account in determining an accurate value of the thermal diffusivity using the laser flash method. This problem can be solved by the following process [2]. First, thermal diffusivity measurements are made for a sample maintained at constant temperature by providing a laser pulse with a different intensity. Next, the experimental values of thermal diffusivity are plotted as a function of the laser pulse intensity. The thermal diffusivity value at the sample temperature before laser irradiation, T_0, should be determined by the extrapolating the curve to the point where the laser intensity is zero. This process is rigorous but appears to be somewhat tedious, so that a simple and useful procedure is strongly needed for practical reasons.

Due to the temperature dependence of the thermal diffusivity and temperature rise of the sample, the value obtained by the laser flash method does not agree with the value at T_0. It is convenient to define an effective temperature rise, T_{eff}, so that the thermal diffusivity at a temperature T_{eff} above T_0 agrees with the measured data. An effective temperature may also be defined as $T_0 + T_{\mathrm{eff}}$. A detailed investigation on T_{eff} has not yet been available, because some complex and lengthy calculation is needed to solve a nonlinear partial differential equation with the temperature dependence of the thermal diffusivity [3]. This paper presents our systematic investigation on the effective temperature rise induced by laser irradiation using a finite difference method and its relevance to the thermal diffusivity value estimated from the measured temperature response curve.

2. THEORETICAL BASIS

Let us consider a finite slab with the uniform temperature distribution of T_0. The thickness and the thermal diffusivity of this slab are denoted by d and α, respectively. A single laser pulse is absorbed uniformly by the front surface of the slab. For this problem, the following thermal diffusion equation (Eq. (1)) should be solved under an initial condition of pulse heating and adiabatic boundary conditions.

$$\frac{\partial T}{\partial t} = \alpha \frac{\partial^2 T}{\partial x^2} \tag{1}$$

where t is the time, x is the distance from the front surface, and T is the temperature.

The finite difference approximation is used to calculate the temperature response. The slab is divided into n elements with no overlapping uniform region with same thickness of Δx. Using the Euler method, Eq. (1) may be converted into the following form [4]. For the internal grids,

$$\frac{T_{i,j+1}-T_{i,j}}{\Delta t}=\frac{\left(\dfrac{T_{i+1,j}-T_{i,j}}{(\frac{1}{2\alpha_{i+1,j}})+(\frac{1}{2\alpha_{i,j}})}\right)-\left(\dfrac{T_{i,j}-T_{i-1,j}}{(\frac{1}{2\alpha_{i,j}})+(\frac{1}{2\alpha_{i-1,j}})}\right)}{\Delta x^2} \tag{2}$$

where index i represents grid location, and index j denotes the time level. Time is indexed such that $t_{j+1}=t_j+\Delta t$ for uniform fixed time step, Δt.

For the back surface of the slab, an adiabatic condition is assumed, and the corresponding equation is given as

$$\frac{T_{n,j+1}-T_{n,j}}{\Delta t}=\frac{-\left(\dfrac{T_{i,j}-T_{n-1,j}}{(\frac{1}{2\alpha_{n,j}})+(\frac{1}{2\alpha_{n-1,j}})}\right)}{\Delta x^2} \tag{3}$$

For the surface irradiated by a laser pulse, the following relation can be given,

$$\frac{T_{1,j+1}-T_{1,j}}{\Delta t}=\frac{\left(\dfrac{T_{2,j}-T_{1,j}}{(\frac{1}{2\alpha_{2,j}})+(\frac{1}{2\alpha_{1,j}})}\right)}{\Delta x^2}+f_j, \tag{4}$$

where f_j represents the intensity of the laser pulse at time level j. Equations (2) to (4) were solved by a FORTRAN90 program with double precision. Δx and Δt are determined so as to satisfy $\alpha\Delta t/\Delta x^2 \leqslant 1/2$ for thermal diffusivity $\alpha_{i,j}$ of any element at any time in order to avoid instability of numerical calculation.

For the calculated temperature response curve, the thermal diffusivity, α, is determined. When the thermal diffusivity of the sample is given by the function of temperature, $\alpha = g(T)$, we can derive an inverse function, $T = g^{-1}(\alpha)$. The effective temperature and effective temperature rise are given by, $g^{-1}(\alpha)$ and $T_{\text{eff}} = g^{-1}(\alpha) - T_0$, respectively.

3. CALCULATION AND RESULTS

3.1. Solution for a Normalized System

The temperature response of the back surface of the sample with constant thermal diffusivity is given in normalized form as follows:

$$T_{\text{norm}} = 1 + 2 \sum_{m=1}^{\infty} (-1)^m \exp(-(m\pi)^2 t_{\text{norm}}) \tag{5}$$

where T_{norm} is the normalized temperature rise of the back surface defined by $(T - T_0)/T_{\text{M}}$ and t_{norm} is the normalized time defined by $t\alpha/d^2$. T_{M} is the maximum temperature rise of the back surface from T_0. This equation shows that the temperature of the back surface reaches half of the maximum temperature rise when the normalized time, t_{norm}, is 0.1388 [1]. The thermal diffusivity, α, is usually estimated from the measured time to reach half of the maximum temperature rise using the following equation:

$$\alpha = 0.1388 d^2 / t_{1/2} \tag{6}$$

where d is the sample thickness.

In order to analyze the temperature response of the sample whose the thermal diffusivity is temperature dependent, some related physical properties are normalized as follows.

$$x_{\text{norm}} = x/d, \quad t_{\text{norm}} = t\alpha_0/d^2, \quad t_{1/2,\,\text{norm}} = t_{1/2}\alpha_0/d^2, \quad T_{\text{norm}} = (T - T_0)/T_{\text{M}},$$
$$\Delta x_{\text{norm}} = \Delta x/d, \quad \Delta t_{\text{norm}} = \Delta t\alpha_0/d^2, \quad \alpha_{\text{norm}} = \alpha/\alpha_0,$$

where α_0 is the thermal diffusivity at the temperature, T_0, before laser heating.

To simplify the expressions, the suffix *norm* indicating a normalized variable is omitted in the following. Equations (1) to (4) can also be applied to derive the normalized temperature response by giving a new meaning of each variable. In the case of $\alpha = 1$, the solution of Eqs. (1) to (4) gives the temperature response of the sample with a temperature-independent thermal diffusivity. When the pulse width represented by f_j in Eq. (4) is sufficiently short, the solution with $\alpha = 1$ provides the normalized temperature rise of Eq. (5).

The linear temperature dependence of thermal diffusivity may be described in the following form with a constant c:

$$\alpha = 1 + cT \tag{7}$$

Here the constant c may be described by $T_M\partial(\alpha/\alpha_0)/\partial T$ in the unnormalized form. This means the thermal diffusivity increases by a factor of 2 when the value of T_M and c are assumed to be 2 K and unity, respectively. It should be kept in mind that thermal diffusivity values of all elements are positive for any time interval in calculations.

Considering these requirements, the temperature response curves were calculated for five cases with $c = -0.01, -0.001, 0.01, 0.1$, and 1.

The laser intensity function, f_i, describing the absorbed laser irradiation by the sample is given in the following form so as to satisfy the condition where T_M is unity and the pulse width is zero (absolute instantaneous heating) for $\Delta t \to 0$.

$$f_j = \begin{cases} n & (j = 1) \\ 0 & (j \neq 1) \end{cases} \tag{8}$$

The calculated temperature response curves are shown in Fig. 1. From the normalized time, $t_{1/2}$, to reach half of the maximum temperature rise, the apparent normalized thermal diffusivity value may be obtained by Eq. (6) as $\alpha = 0.1388/t_{1/2}$. The inverse function of Eq. (7) is $T = (\alpha - 1)/c$. Then, the effective temperature rise, T_{eff}, can be estimated from Eqs. (6) and (7).

$$T_{\text{eff}}/T_M = (0.1388/t_{1/2} - 1)/c \tag{9}$$

The results are summarized in Table I. It can be seen that the uncertainty in the numerical calculation becomes smaller for a larger value of n and

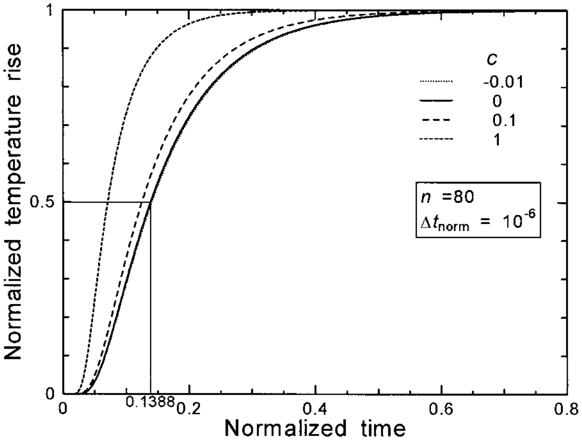

Fig. 1. Normalized temperature response curve showing the linear temperature dependence of thermal diffusivity.

Table I. Normalized Effective Temperature Rise T_{eff}[a]

c	n	Δt	$t_{1/2}$	$T_{\mathrm{eff}}/T_{\mathrm{M}}$
−0.01	40	10^{-4}	0.1403	1.079
	40	10^{-5}	0.1404	1.108
	80	10^{-5}	0.1405	1.194
	80	10^{-6}	0.1405	1.195
−0.001	40	10^{-4}	0.1388	−0.040
	80	10^{-5}	0.1389	0.860
	160	10^{-5}	0.1389	1.002
	320	10^{-6}	0.1389	1.072
	640	10^{-6}	0.1390	1.084
0	10	10^{-4}	0.1371	
	20	10^{-5}	0.1384	
	40	10^{-4}	0.1386	
	40	10^{-5}	0.1387	
	40	10^{-6}	0.1387	
	80	10^{-5}	0.1388	
	80	10^{-6}	0.1388	
	80	10^{-7}	0.1388	
	160	10^{-5}	0.1388	
	160	10^{-6}	0.1388	
	160	10^{-7}	01388	
	320	10^{-6}	0.1388	
	320	10^{-7}	0.1388	
	320	10^{-8}	0.1388	
	640	10^{-6}	0.1388	
	640	10^{-7}	0.1388	
	640	10^{-8}	0.1388	
0.01	40	10^{-4}	0.1370	1.282
	80	10^{-5}	0.1372	1.197
	160	10^{-6}	0.1372	1.183
	320	10^{-6}	0.1372	1.181
	320	10^{-6}	0.1372	1.181
	640	10^{-7}	0.1372	1.181
0.1	40	10^{-5}	0.1251	1.099
	80	10^{-6}	0.1251	1.098
	160	10^{-6}	0.1251	1.098
	320	10^{-7}	0.1251	1.098
	640	10^{-8}	0.1251	1.098
1	10	10^{-4}	0.0715	0.942
	20	10^{-5}	0.0711	0.953
	40	10^{-6}	0.0709	0.959
	80	10^{-6}	0.0708	0.961
	80	10^{-7}	0.0708	0.961
	160	10^{-8}	0.0707	0.962
	320	10^{-8}	0.0707	0.962

[a] In this table, values are normalized as follows: c: $T_{\mathrm{M}}\,\partial(\alpha/\alpha_0)/\partial T$, Δt: $\Delta t\alpha_0/d^2$, $t_{1/2}$: $t_{1/2}\alpha_0/d^2$, T_{eff}: $T_{\mathrm{eff}}/T_{\mathrm{M}}$.

smaller Δt values. The calculations for different values of n and Δt are carried out to evaluate the convergence for a given c. The normalized time for half the maximum temperature rise, $t_{1/2}$, should be 0.1388 for the case where c is zero. The value of $t_{1/2}$ converges asymptotically to a constant value with decreasing Δt and increasing n as shown in the results of Table I.

The present calculation is made for a nonlinear system and, consequently, the normalized values are not identical to those of the different conditions. It is emphasized here that the values of $T_{\text{eff}}/T_{\text{M}}$ are in the range between 1.1 and 1.2 over the wide range of c from -0.01 to 0.1. The temperature rise of the back surface of the sample is known to be several degrees under typical experimental conditions for the laser flash method, and, hence, the absolute value of c is small. For these reasons, it may be safely concluded that the effective temperature rise, T_{eff}, can be approximated by the maximum temperature rise at the back surface of the sample, T_{M}.

3.2. Solution for a Real System

Thermal diffusivities of materials frequently show a nonlinear dependence on temperature, and the laser pulse usually has a finite width. Glassy carbon is considered to be a good example, because its thermal diffusivity is available over a wide temperature range and its nonlinearity is well recognized. For this reason, the temperature response curve of glassy carbon GC-20 [5] was estimated when applying the finite pulse heating conditions.

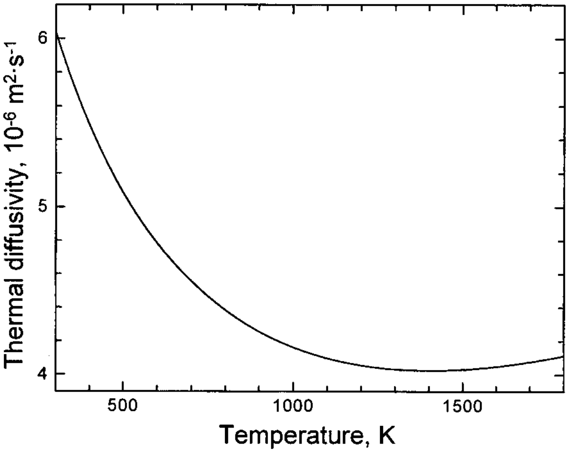

Fig. 2. Thermal diffusivity of glassy carbon GC-20 [5].

Figure 2 shows the thermal diffusivity of glassy carbon of GC-20 measured by the laser flash method. These values have been corrected for the temperature rise effect by the extrapolating to the value where the laser intensity is zero and are represented in the following form as a function of temperature in the temperature range from room temperature to 1800 K.

$$\alpha_{\mathrm{GC}} = 1.239 \times 10^{-9} + 4.226 \times 10^{-3}/(T + 445.8) \tag{10}$$

As can be seen in Fig. 2, the thermal diffusivity of glassy carbon is characterized by a the large negative temperature dependence from 300 to 1000 K and subsequently a region with almost no temperature dependence, as well as a the positive temperature dependence for temperatures higher than 1400 K. To examine such particular features of thermal diffusivity detected for glassy carbon, the temperature response curves were calculated for six cases where three T_0 values are 300, 750, and 1100 K with two T_{M} values of 10 and 2 K. The time interval, Δt, the number of elements, n, and the sample thickness, d, were given by 10^{-6} s, 500, and 4×10^{-3} m, respectively. The laser pulse intensity was assumed constant with a duration of 1 ms. The laser intensity, f_j, is given as follows.

$$f_j = \begin{cases} nT_{\mathrm{M}}/10^3 & (1 \leqslant j \leqslant 10^3) \\ 0 & (10^3 < j) \end{cases} \tag{11}$$

The effective thermal diffusivity, α_{eff}, can be estimated from the calculated temperature response curve using Eq. (12).

$$\alpha_{\mathrm{eff}} = 0.1388 d^2 / t_{1/2} \tag{12}$$

Azumi and Takahashi [6] reported that the effect of the laser pulse width can be excluded when the moment of the intensity of the laser beam as a function of time is set to a starting point of the time. Therefore, the origin of time for the present analysis is set at the center of the laser pulse duration time, i.e., 0.5 ms to calculate $t_{1/2}$ in Eq. (12). Then, the effective temperature rise, $T_{a,\mathrm{eff}}$, is calculated from α_{eff} by assuming $\alpha_{\mathrm{GC}} = \alpha_{\mathrm{eff}}$ at temperature $T_0 + T_{a,\mathrm{eff}}$.

The results are summarized in Table II together with information about the highest sample temperature, $T_{\max}$, obtained in this calculation. The range of the calculations is $T_{\max} < 1800$ K. A difference between $T_{\max}$ and T_0 larger than 500 K is obtained for the case of $T_{\mathrm{M}} = 10$ K. However, it is important to note that the values of $T_{a,\mathrm{eff}}$ are below 12 K for all cases.

Table II. Effective Temperature Rise for Thermal Diffusivity of GC-20

T_0 (K)	T_M (K)	α	$t_{1/2}$ (s)	α_eff ($10^{-6}\,\mathrm{m}^2\cdot\mathrm{s}^{-1}$)	T_max (K)	$T_{a,\,\mathrm{eff}}$ (K)	T_eff (K)
300	2	$\alpha_\mathrm{GC}(T)$	0.3687	6.024	414	2.23	2.3
300	2	$\alpha_\mathrm{GC}(T_0)$	0.3678	6.039	410	-0.11	
300	10	$\alpha_\mathrm{GC}(T)$	0.3722	5.967	920	11.42	11.5
300	10	$\alpha_\mathrm{GC}(T_0)$	0.3678	6.039	850	-0.11	
750	2	$\alpha_\mathrm{GC}(T)$	0.4980	4.460	879	2.04	2.3
750	2	$\alpha_\mathrm{GC}(T_0)$	0.4975	4.464	877	-0.29	
750	10	$\alpha_\mathrm{GC}(T)$	0.4997	4.444	1411	11.18	11.5
750	10	$\alpha_\mathrm{GC}(T_0)$	0.4975	4.464	1386	-0.29	
1100	2	$\alpha_\mathrm{GC}(T)$	0.5422	4.096	1233	1.46	2.3
1100	2	$\alpha_\mathrm{GC}(T_0)$	0.5420	4.097	1232	-0.87	
1100	10	$\alpha_\mathrm{GC}(T)$	0.5428	4.091	1766	10.38	11.2
1100	10	$\alpha_\mathrm{GC}(T_0)$	0.5420	4.097	1762	-0.87	

In this work, the temperature response curves are also calculated using the values of thermal diffusivities of α_GC at T_0, $\alpha_\mathrm{GC}(T_0)$ under the same condition. This corresponds to the case where the temperature dependence is not considered. The effective temperature rise for this case is represented by the value for $\alpha = \alpha_\mathrm{GC}(T_0)$. It may be noteworthy that the value of T_eff for $\alpha_\mathrm{GC}(T_0)$ should be zero when the numerical error is considered to be negligibly small. On the other hand, the absolute value of the apparent effective temperature rise derived by the calculation, $T_{a,\,\mathrm{eff}}$, for $\alpha_\mathrm{GC}(T_0)$ becomes larger at a temperature T_0 above 1000 K. This is attributed mainly to the relatively small value of the temperature dependence of glassy carbon in that region. With these facts in mind, we introduce the temperature difference between the apparent effective temperature rise for $\alpha_\mathrm{GC}(T_0)$ and that of the actual temperature dependence, $\alpha_\mathrm{GC}(T)$, by providing an expression for the effective temperature rise T_eff.

This effective temperature rise, T_eff, lies in the range between $1.1T_\mathrm{M}$ and $1.2T_\mathrm{M}$. These results undoubtedly agree well for the case where a linear temperature dependence of thermal diffusivity is assumed. As shown in Fig. 2, the nonlinearity of thermal diffusivity for glassy carbon is well recognized. However, the present results clearly suggest that the effect of such nonlinearity is not so significant in estimating the thermal diffusivity value from the measured temperature response using the laser flash method. This is because the high temperature rise occurs in the very limited time and space region only. Therefore, we could use a rather simple method for which the maximum temperature rise, T_M, at the back surface of the sample is considered to be equal to the effective temperature rise, T_eff.

4. CONCLUDING REMARKS

The effective temperature rise has been estimated by the finite difference method in order to determine the sample temperature corresponding to the thermal diffusivity measurement using the laser flash method. The calculation was also made in the case of glassy carbon whose thermal diffusivity shows a nonlinear temperature dependence. In conclusion, the effective temperature rise T_{eff} induced by laser irradiation is well approximated by a value of about 1.1 to 1.2 times that of T_{M}, where T_{M} is the maximum temperature rise of the back surface of the sample. Considering that T_{M} is several degrees in the typical experimental conditions, we could use the useful and simple relation that the effective sample temperature is given by $T_0 + T_{\mathrm{M}}$ in the thermal diffusivity measurement by the laser flash method.

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