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Determination of the Thermal Diffusivity and Specific Heat Using an Exponential Heat Pulse, Including Heat-Loss Effects[1]

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The one-dimensional heat diffusion equation has been solved analytically for the case of a heat pulse of the form $F(t) = \exp(-t/\tau)/\tau$ applied to the front face of a homogeneous body including the effects of heat loss from the front and back faces. Approximate expressions are presented which yield a simple, accurate technique for the determination of the thermal diffusivity and specific heat, suitable to a wide range of heat-pulse time constant and heat-loss parameters, without recourse to graphical techniques or requiring further computer analysis. A procedure is described for the determination of an effective time constant to allow application of the present results to the case of a nonexponential heat pulse. Experimental results supporting the theoretical analysis are presented for five samples of silicon germanium alloys of various thicknesses, determined using a xenon flash tube heat-pulse exhibiting an exponential dependence. Proper consideration of the experimental heat pulse shape is shown to lead to reliable corrections to the apparent thermal diffusivity, even for relatively long heat-pulse times.

KEY WORDS: heat capacity; heat-loss correction; heat-pulse method; specific heat; thermal diffusivity.

1. INTRODUCTION

In the flash method for measuring thermal diffusivity [1] a pulse of radiant energy is deposited on the front face of a small, thin sample and the temperature history of the back face is monitored as a function of time. The

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technique has been extended [2–8] to account for heat losses (when the temperature of the back face of the sample reaches a maximum and then decreases with further time) and nonzero duration of the heat pulse assuming various pulse shapes [4, 9, 10], which tends to delay the temperature response of the back face to the heat pulse. The subject of this report is that of a pure exponential heat pulse, which has not been previously presented. Relying heavily on the work of previous authors, the one-dimensional heat diffusion equation is solved analytically and numerically for the case of a pure exponential heat pulse including the effects of heat loss.

2. THEORY

When the duration of the heat pulse represents a significant fraction of the transit time for an isothermal temperature front across a sample, the response of the back face of the sample will be delayed somewhat compared to the response due to an instantaneous heat pulse. At high temperatures radiation losses inevitably become significant, in addition to heat losses due to conduction down sample supports, requiring that heat losses also be taken into account. The heat diffusion equation for these boundary conditions will now be solved exactly, assuming a simple exponential heat pulse.

Consider a flat slab with parallel faces at $x = 0$ and $x = d$ and infinite lateral extent. The solution to the one-dimensional heat diffusion equation for the time dependence of the temperature of the back face, assuming an instantaneous planar heat pulse at $t = 0$, $x = 0$, and equal heat-loss parameters (a linear function of the temperature difference from the surroundings, denoted by L) describing the two surfaces, is, following Watt [6],

$$T(t/t_c, L)/T_\infty = 2 \sum_{n=0}^{\infty} \frac{\beta_n \cos(\beta_n) + L \sin(\beta_n)}{\beta_n^2 + L^2 + 2L} \beta_n e^{-\beta_n^2 t/t_c} \qquad (1)$$

where β_n are the positive roots of

$$(\beta^2 - L^2) \tan(\beta) = 2L\beta \qquad (2)$$

the material parameters are represented by $t_c = d^2/\alpha$, and T_∞ is the final temperature in the absence of heat losses. For the exponential heat pulse such as shown in Fig. 1 with the analytic form (for a unit pulse)

$$F(t) = \begin{cases} 0, & t < 0 \\ \frac{1}{\tau} e^{-t/\tau}, & t \geq 0 \end{cases} \qquad (3)$$

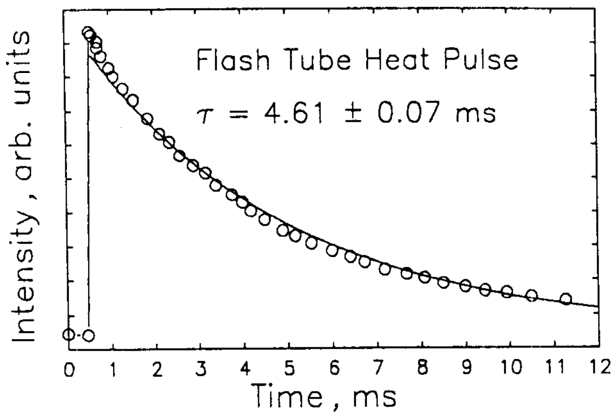

Fig. 1. Heat-pulse function as recorded by a photodiode detector for a commercial xenon flash tube. The solid line indicates the simple exponential behavior with $\tau = 4.61 \pm 0.07$ ms.

solutions are obtained, following Heckman [7], by evaluating the integrals

$$T(t/t_c, L, \tau/t_c)/T_\infty = \int_{-\infty}^{t} F(t')\, G\left(\frac{t-t'}{t_c}, L\right) dt' \tag{4}$$

where G is the solution of the instantaneous heat pulse problem given by Eqs. (1) and (2).

The integrations are readily performed, yielding

$$T(t/t_c, L, \tau/t_c)/T_\infty = 2 \sum_{n=0}^{\infty} \frac{\beta_n \cos(\beta_n) + L \sin(\beta_n)}{\beta_n^2 + L^2 + 2L}\, \beta_n \left[\frac{e^{-\beta_n^2 t/t_c} - e^{-t/\tau}}{1 - \beta_n^2 \tau/t_c}\right] \tag{5}$$

which reduces to Eq. (1) for $\tau = 0$, as it must. If τ/t_c is sufficiently small and t/τ is sufficiently large, the term in brackets may be approximated by $\exp[-\beta_n^2(t-\tau)/t_c]$, which implies that the time dependence of the temperature variation of the back face for an exponential heat pulse is nearly identical to the instantaneous heat-pulse case simply delayed by the time τ, i.e.,

$$T(t/t_c, L, \tau/t_c)/T_\infty \cong T\left(\frac{t-\tau}{t_c}, L, 0\right) \tag{6}$$

This indicates that heat-loss correction methods which neglect finite pulse effects (such as those due to Cowan [5] or Clark and Taylor [8]) can be extended to the case of an exponential pulse simply by replacing any time which occurs in the former analysis, such as $t_{0.5}$ or $5t_{0.5}$ with the experimentally observed times reduced by the amount τ.

For negligible heat losses and small τ/t_c, for example, the thermal diffusivity is given by

$$(t_{0.5}-\tau)=0.13875\ t_c=0.13875\ d^2/\alpha \qquad (7)$$

which implies that neglect of the finite pulse effect will result in a fractional underestimate in α of the order $\tau/t_{0.5}$. If heat loss can be ignored, a plot of $t_{0.5}$ vs d^2 will then yield an estimate of τ as the intercept and α can be determined from the slope. Indeed, determining τ in this manner for a particular heat pulse source should yield reasonable estimates for α even for pulse shapes deviating significantly from an exponential since the delayed-time response of the back face, indicated in Eq. (6), appears to be a general future of finite pulse effects.

Parker has suggested [11] accounting for finite pulse effects by measuring $t_{0.5}$ not from the onset of the pulse but from the time at which half the energy of the pulse is deposited in the sample. Larson and Koyama (see Ref. 10, Eqs. 43–45) also find expressions very similar to Eq. (7), suggesting a more broad range of validity once the appropriate τ has been determined for a particular pulse shape. Based on Eqs. (5)–(7), the correct value of τ for a nonexponential pulse might also be estimated as the time required to deposit ln(2) (not $\frac{1}{2}$) of the total heat in the sample. This method may be more reliable than the $t_{0.5}$ vs d^2 method described above because the latter method can be distorted by heat loss effects. Utilizing an incorrect estimate for τ will result in an error in α nearly as large as a neglect of τ itself and is therefore not inconsequential.

3. NUMERICAL RESULTS

In the absence of heat loss ($L=0$) only one experimental data point on the temperature history of the back face is required to uniquely determine α, assuming that τ is known. A convenient point is $t_{0.5}$. The ratio $t_{0.5}/t_c$ has been calculated as a function of τ/t_c and is shown in Fig. 2. The dashed line represents the approximation in Eq. (7), which yields a maximum error of about 2% for $\tau/t_c=0.1$ or $\tau/t_{0.5}=0.7$. Thus, Eq. (7) should be useful for pulse times nearly as large as $t_{0.5}$ itself.

A minimum of two experimental data points on the temperature history of the back face is required to uniquely determine α and L, assuming that τ is known. Two particularly easy points to determine are $t_{0.5}$ and t_m. The ratio T_m/T_∞ and $t_{0.5}/t_c$ can then be parametrized as a function of

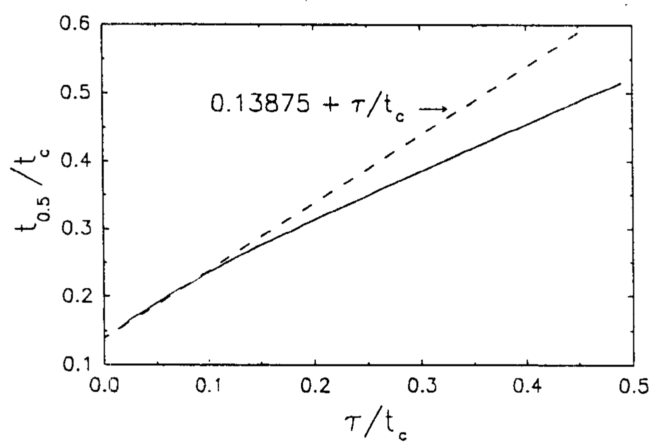

Fig. 2. The effect of an exponential heat pulse on the half-time, with no heat loss. The dashed line represents the first-order approximation useful for small τ.

$t_{0.5}/t_m$. Equation (5) has been solved numerically for values of L between 0 and 1, with $\tau = 0$, the results of which are shown in Fig. 3. The curves in Fig. 3 have been fit to simple expressions yielding a useful ($\tau = 0$) interpolating formula for the thermal diffusivity and maximum temperature:

$$\alpha^{a}/d^{2} = 0.13875\,(1 - e^{1.8073 - 1.2407x})/y \tag{8}$$

$$T_{m}^{a}/T_{\infty} = 1 - e^{2.608 - 1.2841x} \tag{9}$$

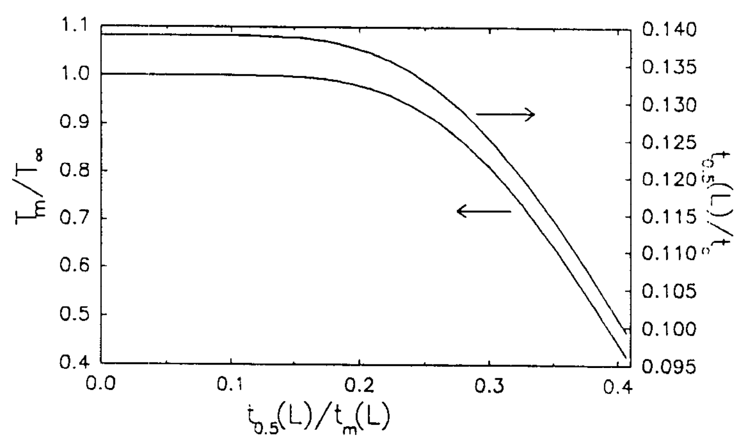

Fig. 3. Heat-loss effect on the maximum temperature and half-time parameterized by the ratio t_c/t_m.

with $x = t_m/t_{0.5}$ and $y = t_{0.5}$. The superscript a is intended to indicate that these expressions represent estimates of the full solutions to Eq. (5).

From the above discussion we expect Eqs. (8) and (9) to be useful for nonzero τ if instead we use $x = (t_m - \tau)/(t_{0.5} - \tau)$ and $y = t_{0.5} - \tau$. The fractional error between Eq. (8) and the direct numerical solution of Eq. (5) for $0.00 \leqslant \tau/t_c \leqslant 0.05$ is shown in Fig. 4, which may be used to estimate the corrections to Eq. (8), if required. Note that the several curves in Fig. 4 represent different values of τ/t_c, not the more readily determined experimental parameter $\tau/t_{0.5}$. A good first-order estimate of τ/t_c is given by $0.139\,\tau/t_{0.5}$. Even for a relatively large value of $\tau/t_{0.5} = 0.36$ ($\tau/t_c = 0.05$), Eq. (8) is in error by only a few percent, which can easily be estimated to sufficient accuracy from Fig. 3 if required. Equation (8) will be accurate to better than 0.4% even for very large heat losses when $\tau/t_{0.5} < 0.1$.

The specific heat may be calculated exactly from

$$C = \frac{Q}{T_\infty} = \frac{Q}{T_m}(T_m/T_\infty) \tag{10}$$

or estimated from

$$C^a = \frac{Q}{T_m}(T^a_m/T_\infty) \tag{11}$$

where Q and T_m are the experimental quantities and T^a_m/T_∞ is given by Eq. (9). Figure 5 shows the error in using Eqs. (9) and (11) to estimate the specific heat.

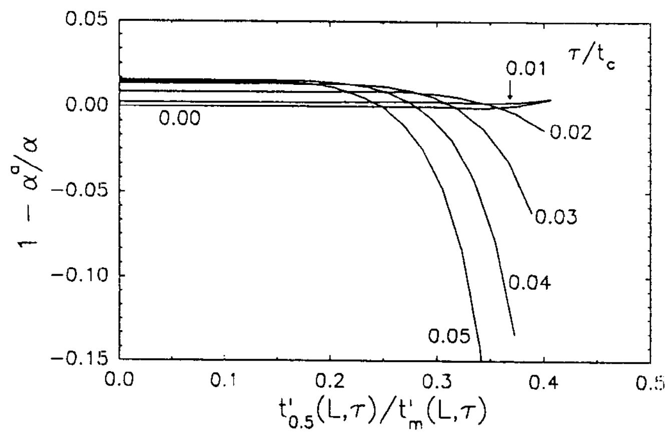

Fig. 4. The fractional error in Eq. (8) for the thermal diffusivity as a function of heat-loss and heat-pulse effects. The primes indicate that the observed times should be reduced by the amount τ.

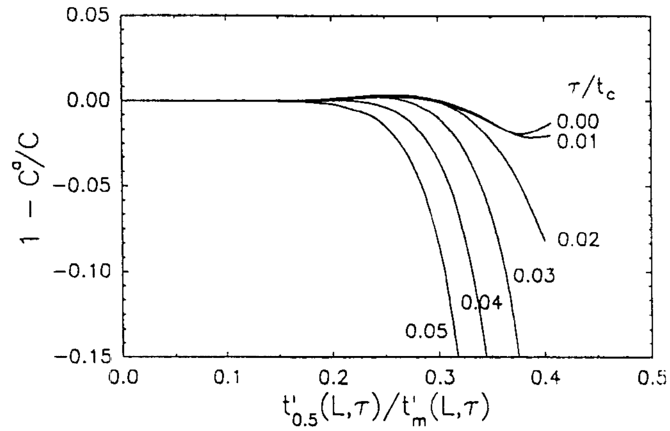

Fig. 5. The fractional error in Eqs. (9) and (11) for the specific heat as a function of heat-loss and heat-pulse effects. The primes indicate that the observed times should be reduced by the amount τ.

Equations (8) and (9) have also been compared to the appropriate values in Heckman's tables [7] and found to agree to better than 1% up to $L = 1$. Thus, Eqs. (8), (9), and (11) may be used to calculate corrections to the diffusivity and specific heat due to heat-pulse and heat-loss effects with a high accuracy, without recourse to tables, graphs, or the full analytical solution represented by Eqs. (2) and (5).

4. EXPERIMENTAL RESULTS

The thermal diffusivity is determined from 600 to 1300 K using an apparatus described in detail elsewhere [12, 13]. A commercial xenon flash tube applies a heat pulse to the front surface of the sample of thickness d by means of a sapphire light pipe. The sample holder consists of four equally spaced rods (alumina tubes over tungsten wire), designed to reliably position each sample 0.001 m from the end of the light pipe. The front and back sample surfaces are sputtered with graphite to provide a reproducible emissivity for each sample. Figure 1 indicates a typical heat pulse from the xenon flash tube. The pulse is reasonably well approximated by a simple exponential with a time constant $\tau = 4.61 \pm 0.07$ ms.

An InSb infrared detector determines the temperature of the back face of the sample. The output of the detector is amplified using a Tektronix differential amplifier and displayed on a Nicolet digital storage oscilloscope. The values of $t_{0.5}$ (the time required for the temperature of the back face to first reach one-half of the maximum value), t_m (the time required for the

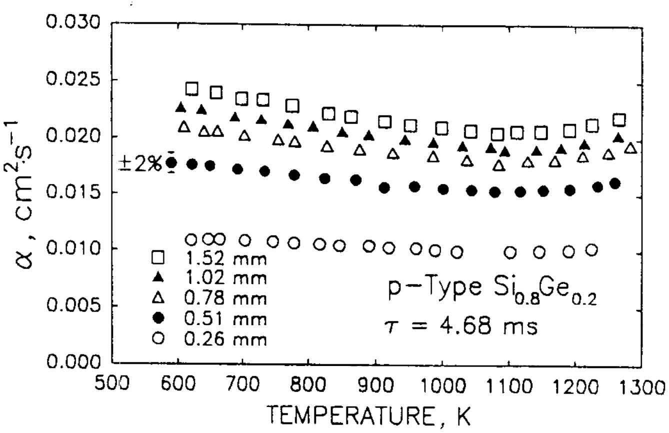

Fig. 6. The apparent thermal diffusivity of five samples of *p*-type $Si_{0.8}Ge_{0.2}$ as a function of temperature, neglecting heat-loss and heat-pulse effects.

temperature of the back face to reach the maximum value), and T_m (the maximum temperature of the back face) are determined directly from the oscilloscope display, utilizing a calibration curve to convert the detector output voltages to temperatures. The lamp is flashed three or four times at each temperature with a typical reproducibility of $\pm 0.5\%$ for T_m. The total heat, Q, deposited on the front face of the sample may be determined using a sample of known specific heat.

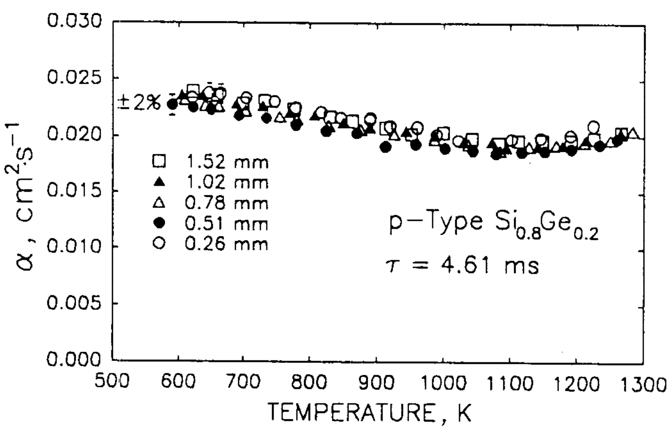

Fig. 7. The thermal diffusivity of five samples of $Si_{0.8}Ge_{0.2}$ as a function of temperature, including heat-loss and heat-pulse effects as described by Eq. (8).

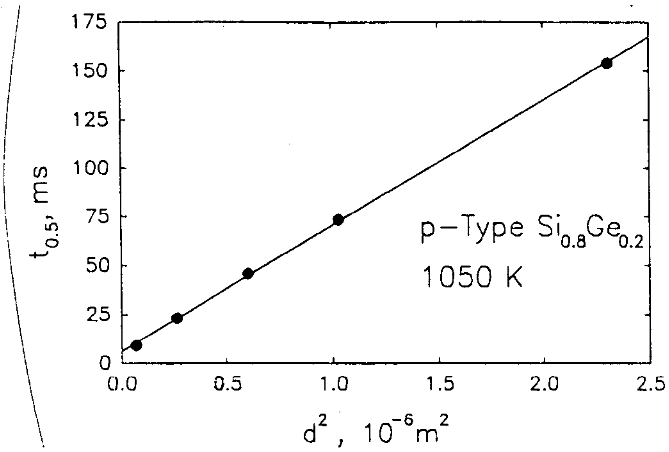

Fig. 8. The half-time as a function of thickness for five samples of $Si_{0.8}Ge_{0.2}$. The indicated intercept overestimates the actual τ by 50% due to heat-loss effects.

The diffusivity is calculated from d, τ, $t_{0.5}$, and t_m using the theory described above. Since only times are involved, the determination of the diffusivity is relatively insensitive to experimental details. Determination of the specific heat, however, requires determinations of T_m and Q using calibrations which are critically sensitive to experimental details such as sample and detector positions, surface preparation, and heat-pulse reproducibility [14].

Figure 6 shows the uncorrected thermal diffusivity, $\alpha = 0.13875 \circ d^2/t_{0.5}$, as a function of temperature for five samples of p-type $Si_{0.8}Ge_{0.2}$, calculated neglecting heat-loss and finite pulse effects. Figure 7 shows the same results after correction for heat-loss and finite pulse effects using Eq. 8 above. The scatter in the corrected data is of the order of $\pm 2\%$, which represents excellent agreement.

Figure 8 shows a typical plot of $t_{0.5}$ vs d^2 for these five samples. The nonzero intercept representing the effect of the finite pulse time is in clear evidence. A fit to this line yields an estimate of $\tau = 7.09$ ms, which is considerably larger than determined from the actual heat pulse in Fig. 1. The data in Fig. 8, however, include the effect of heat loss, which tends to lower the observed $t_{0.5}$, particularly for the thickest samples. This method therefore, overestimates τ by about 50% in this case. For greatest accuracy, τ should be determined directly from the heat pulse, as was done in Fig. 1 and used for the calculation in Fig. 7.

6. CONCLUSION

A technique for determining the thermal diffusivity and specific heat from the time to reach the maximum temperature and the time to reach half the maximum temperature has been presented, which culminates in the simple expressions given in Eqs. (8), (9), and (11) above. The technique described here is consistent with previous attempts to correct for heat-pulse and heat-loss effects, but is simpler to apply since no tables or graphs are required to reduce the data. Moreover, the present method accounts for the shape of the actual heat pulse employed and unambiguously identifies τ as the time required for the pulse to deposit ln(2) of the total heat onto the front face of the sample. The validity of Eq. (8) for the diffusivity has been experimentally confirmed to within $\pm 2\%$ by determining the thermal diffusivity on several samples with various thicknesses.

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