SAND-77-0973

CONF-770847-5

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THERMAL DIFFUSIVITY MEASUREMENT OF TEMPERATURE SENSITIVE MATERIALS
BY AN EXTENDED PULSE TECHNIQUE¹

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Difficulty arises in applying the standard flash techniques for measuring thermal diffusivity to temperature sensitive materials because decomposition or ignition frequently results from the high front surface temperature. This difficulty can be overcome by simultaneously reducing the intensity of the energy source and increasing the exposure time. The increased exposure time permits a sufficient amount of energy to propagate to the opposite surface of the specimen so that a detectable temperature excursion results.

This paper discusses a theoretical analysis of the problem and includes extended pulse effects, radial heat conduction effects, and surface heat loss effects. The results are used to consider (1) the regime of pulse times for which data reduction can be based on the instantaneous pulse model, (2) the regime of specimen diameter/thickness ratios for which radial conduction effects can be neglected (this consideration allows minimizing explosive mass and hence, the hazard) and (3) the estimation of the front surface temperature during heating (so that ignition or decomposition temperatures are not reached).

Also, the results of experiments with the explosives HNS and RDX are discussed. Data reduction is made by finding the values of thermal diffusivity and surface heat loss coefficients which minimize the sum of the squares deviation between the experimental response curve and the theoretical response curve.

¹ This work was supported by the United States Department of Energy

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Theoretical Analysis

The geometry which will be considered is that of a disk (sample) which is assumed to be well clamped on the circumference to a sample holder. The energy fluence to the sample is assumed to be constant for the time interval $0 \leq t \leq \hat{t}$ and uniform with respect to radial and angular position. The energy equation and boundary and initial conditions for this problem are

Energy Equation

$$\nabla^2 T(z, r, t) = \frac{1}{\alpha} \frac{\partial T}{\partial t}(z, r, t)$$

Boundary Conditions

1. $-\frac{\partial T}{\partial z}(0, r, t) = f(t) - \frac{h_1}{\lambda} [T(0, r, t) - T_s]$
2. $-\frac{\partial T}{\partial z}(\ell, r, t) = \frac{h_2}{\lambda} [T(\ell, r, t) - T_s]$
3. $\frac{\partial T}{\partial r}(z, 0, t) = 0$
4. $T(z, R, t) = T_s$

Initial Condition

$$T(z, r, 0) = T_s$$

$$\text{where } f(t) = \begin{cases} 0 & t < 0 \\ Q/\lambda A & 0 \leq t \leq \hat{t} \\ 0 & t > \hat{t} \end{cases}$$

The solution of this problem for $\hat{t} \rightarrow 0$ has been given by Watt [1] and can be written in terms of nondimensional parameters as

$$\theta(\xi, \eta, \tau) = \left\{ \frac{2}{\sigma} \sum_{m=1}^{\infty} \frac{J_0(\beta_m \sigma \eta) e^{-\frac{\beta_m^2}{\sigma^2} \tau}}{\beta_m J_1(\beta_m \sigma)} \right\}.$$

$$\left\{ C + 2 \sum_{n=1}^{\infty} \frac{\gamma_n (\gamma_n^2 + H_2^2) [\gamma_n \cos(\gamma_n \xi) + H_1 \sin(\lambda_n \xi)] e^{-\gamma_n^2 \tau}}{(\gamma_n^2 + H_1^2)(\gamma_n^2 + H_2^2 + H_1) + H_1(\gamma_n^2 + H_2^2)} \right\}$$

where $C = \begin{cases} 0 & \text{if } H_1 \text{ or } H_2 \neq 0 \\ 1 & \text{if } H_1 = H_2 = 0 \end{cases}$

and the eigenvalues β_m and γ_n are determined from the eigenequations

$$J_1(\beta_m \sigma) = 0$$

and

$$\tan(\gamma_n) = \frac{\gamma_n(H_1 + H_2)}{\gamma_n^2 - H_1 H_2},$$

respectively. This solution can now be generalized to solve the case for $\hat{t} > 0$ by Duhamel's theorem:

$$\phi(\xi, \eta, \tau) = \int_{s=0}^{\tau} f(s) \cdot \frac{\partial \theta}{\partial \tau}(\xi, \eta, \tau - s) ds$$

Although this solution is all that is needed for reduction of experimental data, three special cases will first be considered which have a bearing on the experiment.

First, since the sample material is temperature sensitive, an estimate of the maximum temperature is useful so that degradation or ignition of the sample can be avoided. Rather than using the complete solution, an upper estimate can be obtained from the solution for the problem of a semi-infinite solid which is exposed to a constant heat flux. From Carslaw & Jaeger [2], the surface temperature rise will be

$$\Delta T(0, \hat{t}) = 2(Q/\lambda) \cdot \sqrt{\frac{\alpha \hat{t}}{\pi}}$$

where surface heat loss has been neglected. Use of this equation requires an estimate of the magnitude of α and λ .

Second, the effect of finite pulse time on the half-time, $\tau_{1/2}$ [3], can be computed from the general solution. The case which is treated is that of a semi-infinite slab ($\sigma = \infty$) so that radial conduction is neglected and the nondimensional surface loss coefficients for both front and rear surfaces are equal. Results are shown in Figure 1 and indicate that if a half-time deviation of less than 1% is desired, then the pulse width, $\tau_{1/2}$, should be less than ~ 0.004 .

Third, the effect of variation in the sample radius-to-thickness ratio on half-time can be computed from the general solution. Figure 2 shows this result for zero pulse width and indicates that if data reduction is based solely on half-time, then σ should be greater than ~ 2.2 in order to eliminate radial conduction effects.

Figure 1 Half-time deviation from value for $\hat{\tau}=0$ as a function of $\hat{\tau}$, all for $\sigma=\infty$

Figure 2 Half-time deviation from value for $\sigma=\infty$ as a function of σ , all for $\hat{\tau}=0$

Experiment

Figure 3 illustrates the experimental configuration. A 750 mW laser is used as the power source, and a camera shutter is used to adjust the pulse width. A differential thermocouple at the rear center of the sample and the sample holder is used to measure temperature excursion. A temperature controller is used to maintain the sample at a preset temperature. The sample and sample holder environment can be evacuated, and the sample is exposed on a downward facing surface--both considerations to minimize convective heat loss.

Figure 3 Schematic of apparatus

The experimental temperature response on the rear surface is recorded on an analog recorder. Data reduction is accomplished by a computer program that finds the values of α , H_1 and H_2 which minimize the sum-of-the squares deviation between the general solution and the data at specified data points.

Figures 4 and 5 show the experimental data, the fitted solution and the input/output parameters for the explosives HNS (hexanitrostilbene) and RDX (trinitrotriazacyclohexane), respectively. Also shown is an estimate of the maximum front surface temperature for the HNS experiment and a nondimensional pulse width for the RDX experiment.

Figure 4 Experimental and theoretical response for HNS

Figure 5 Experimental and theoretical response for RDX

Conclusions

The extended pulse technique appears to be a useful way of applying the flash method to measure the thermal diffusivity of temperature sensitive materials. By properly designing an experiment, temperature excursions can be made sufficiently small, finite pulse width effects on data reduction can be minimized, and the sample diameter can be reduced to the smallest value for which radial conduction is inconsequential.

Nomenclature

H_1, H_2	front and rear surface Nusselt numbers
h_1, h_2	front and rear surface linear heat loss coefficients
J_0, J_1	ordinary Bessel functions of zeroth and first order
l	sample thickness
Q/A	heat flux
R	sample radius
r	radial position variable
s	dummy integration variable
T	temperature
T_s	surrounding temperature
t	time variable
$\hat{t}$	pulse width
z	axial position variable
α	thermal diffusivity
η	nondimensional radial position variable ($\equiv r/R$)
θ	nondimensional temperature excess for zero pulse width [$\equiv (T - T_s)/(Ql/\lambda A)$]
λ	thermal conductivity
ξ	nondimensional axial position variable ($\equiv z/l$)

- σ ratio of sample radius to thickness ($\equiv R/\ell$)
- τ Fourier number ($\equiv \alpha t/\ell^2$)
- $\hat{t}$ nondimensional pulse width ($\equiv \alpha \hat{t}/\ell^2$)
- ϕ nondimensional temperature excess for finite pulse width
 $[\equiv (T - T_s)/(Q\ell/\lambda A)]$

References

1. Watt, D. A., Br. J. Appl. Phys., 17, 231 (1966).
2. Carslaw, H. S. and Jaeger, J. C., Conduction of Heat in Solids, 2nd ed., Oxford Press, London, p. 75.
3. Half-time is customarily used in data reduction, see for example Parker, W. J., et al, J. Appl. Phys., 32 1679 (1961).