# Multidimensional Flash Diffusivity Measurements of Orthotropic Materials

S. Graham,<sup>1</sup> D. L. McDowell,<sup>1, 2</sup> and R. B. Dinwiddie<sup>3</sup>

Received July 14, 1998

A generalization of the radial flash technique is presented whereby the thermal diffusivity of an orthotropic solid is measured in directions parallel and perpendicular to the flash source. The theoretical formulation is based on a Green's function approach which assumes a general orthotropic solid with three mutually orthogonal thermal diffusivities (or conductivities). Using this approach, a solution to this problem is presented which can be used to develop solutions for arbitrary pulse waveforms and incident geometries. Analytical and numerical results are presented for two-dimensional and three-dimensional cases of finite and semiinfinite solids. Characteristic equations which describe the ratio of the temperatures at two points along a principal axis are given. The equations show excellent agreement with numerical predictions as well as experimental results. A parameter estimation approach is given which improves on the accuracy of the radial flash technique in the determination of thermal diffusivity from experimental data.

**KEY WORDS:** Composites; flash method; orthotropic; thermal conductivity; thermal diffusivity; transient technique.

# **1. INTRODUCTION**

Numerous techniques have been developed to measure thermal conductivity through both steady-state and transient schemes (cf. [1, 2]). Among the most widely employed methods for planar specimens is an indirect technique called the flash.diffusivity test [3]. In its most basic form, the

<sup>&</sup>lt;sup>1</sup>Woodruff School of Mechanical Engineering, Georgia Institute of Technology, Atlanta, Georgia 30332-0405, U.S.A.

<sup>&</sup>lt;sup>2</sup> To whom correspondence should be addressed.

<sup>&</sup>lt;sup>3</sup> Oak Ridge National Laboratory, Oak Ridge, Tennessee 37831, U.S.A.

<sup>0195-928</sup>X/99/0300-0691\$16.00/0 (C 1999 Plenum Publishing Corporation

flash diffusivity technique correlates a simple thermal response model for isotropic, homogeneous materials with experimental data. Once the thermal diffusivity is obtained from the semianalytical fit, the thermal conductivity is calculated. Since it is idealistic to assume that all of the boundary conditions of the Parker technique will be met in a laboratory situation, many corrections to the Parker analysis have been proposed for finite-pulse time effects, heat losses, and nonuniform irradiation [4–9].

In cases of nonuniform heating, multidimensional heat flow effects are induced in the material sample. Donaldson [10, 11] investigated an extreme case of nonuniform heating in which only a small spot of the surface of the sample was irradiated. This method was later used by Donaldson and Taylor [12] to determine the thermal diffusivity in the radial direction by taking the ratio of the temperatures at two points on the back surface. This technique is known as the radial flash technique. This procedure was the first extension of the Parker method which allowed the determination of the thermal diffusivity of materials in directions perpendicular to the axis of heat impulse. This data reduction scheme was also shown to be unaffected by heat losses on the front and rear surfaces [13], independent of the heat absorbed, and required no destructive sectioning to obtain material samples. However, this technique was developed for isotropic materials and does not fully describe the temperature response of orthotropic materials. It has also been documented that the radial flash method is not as accurate as Parker-based methods due to the sensitivity of the results on a knowledge of the distance between the two surface temperatures [14]. The Parker flash technique may be applied to orthotropic materials by destructive sectioning of material samples perpendicular to the suspected principal axes of heat flow. Since the radial flash technique is a nondestructive approach, it offers some desirable advantages over unidirectional techniques provided the accuracy can be improved. Variations of the radial flash technique were employed in the measurement of the thermal conductivity of thin films [15–18] and anisotropic materials [14, 19]. In the measurement of thin films, the assumption of one-dimensional heat flow along the length of the film is used to obtain the thermal diffusivity along that path without regard for anisotropy. In the measurement of orthotropic materials, it is assumed that the energy flash occurs on a transversely isotropic plane which is a special case of orthotropy. Due to this assumption, this procedure is applicable in only a limited sense to materials which exhibit full orthotropic behavior by measuring only along principal directions. More general considerations are important when applying this procedure to heterogeneous materials which possess orthotropic symmetry, textured metals, laminated composites, and other materials possessing an appropriately oriented microstructure.

This paper presents the general solution to the differential equation which describes the three-dimensional heat flow in a homogeneous orthotropic slab subjected to an instantaneous heat pulse. In this analysis, no plane of transverse isotropy is assumed. By treating such a case, a generalized in-plane method is given which can be used for arbitrary pulse geometries and waveforms. Both semiinfinite and finite geometries are treated, and their implications for experimental procedures are discussed. Correlation of the solution with numerical simulation and experimental data is presented, demonstrating the validity of the procedure.

# 2. ANALYSIS

Consider a homogeneous orthotropic solid which is exposed to any general thermal boundary condition. The governing heat conduction equation which describes the temperature distribution in the solid for this situation is given by

$$K_x \frac{\partial^2 T}{\partial x^2} + K_y \frac{\partial^2 T}{\partial y^2} + K_z \frac{\partial^2 T}{\partial z^2} + g = \rho c \frac{\partial T}{\partial t}$$
(1)

Here g is a volumetric energy generation term,  $\rho$  is the density, c is the specific heat of the material, and  $K_i$  (i = x, y, z) are the mutually orthogonal thermal conductivities. This differential equation may also be applied to heterogeneous materials provided that the scale of the heterogeneity is sufficiently small compared to the sample size [20-23]. In such a situation, we will assume that volume averaged quantities sufficiently describe the macroscopic material behavior. We may simplify the solution to this differential equation by performing the following coordinate transformation [24],

$$x' = x \left(\frac{K}{K_x}\right)^{1/2}, \qquad y' = y \left(\frac{K}{K_y}\right)^{1/2}, \qquad z' = z \left(\frac{K}{K_z}\right)^{1/2}$$
 (2)

where K is the effective thermal conductivity given by  $K = (K_x K_y K_z)^{1/3}$ . This results in the following governing differential equation:

$$K\left(\frac{\partial^2 T}{\partial x'^2} + \frac{\partial^2 T}{\partial y'^2} + \frac{\partial^2 T}{\partial z'^2}\right) + g = \rho c \frac{\partial T}{\partial t}$$
(3)

Equation (3) is now equivalent to the differential equation for a homogeneous isotropic material in a space which is distorted from the original. This equation can now be solved using any number of standard techniques applicable to homogeneous isotropic materials and transformed back to the original space to obtain the solution. In the following analysis, we employ a Green's function approach to determine the temperature distribution in a solid subjected to a uniform instantaneous flash on one surface. The foundation of the following approach may be found in several sources [24, 25] and is presented for a general understanding of the results.

# 2.1. A Solid of Finite Geometry

Consider a homogeneous orthotropic slab with an instantaneous surface heat source over a symmetric planar region and having a uniform initial temperature (t=0) of zero as shown in Fig. 1. Inherent in this analysis is the assumption that the geometric axes are aligned with the



Fig. 1. (a) Three-dimensional orthotropic slab with incident heat pulse and (b) quarter-symmetry solution domain; the flash region is the darkened square.

three principal coordinate directions of heat flow. In addition, adiabatic conditions are assumed to exist on all exterior boundaries. Due to the symmetry of the problem, only the-quarter portion of the slab depicted in Fig. 1b need be analyzed. The boundary conditions for this problem in the transformed coordinate system are

$$\frac{\partial T}{\partial x'} = 0; \qquad x' = 0, \qquad x' = a'$$
 (4)

$$\frac{\partial T}{\partial y'} = 0; \qquad y' = 0, \qquad y' = b' \tag{5}$$

$$\frac{\partial T}{\partial z'} = 0; \qquad z' = 0, \qquad z' = L' \tag{6}$$

$$T = 0; \qquad t = 0 \tag{7}$$

Due to the uniform initial temperature of zero and homogeneous boundary conditions, the temperature distribution in the slab for t > 0 due to a single point source may be determined by the integral [24],

$$T(x', y', z', t) = \frac{1}{\rho c} \int_{\tau=0}^{t} \int_{V} G(x', y', z', t \mid x'', y'', z'', \tau) g(x'', y'', z'', \tau) dV d\tau$$
(8)

where G represents the Green's function for this problem and g is the energy generation due to an instantaneous point source at  $x'' = \zeta'$ ,  $y'' = \eta'$ , and z'' = 0 in the transformed coordinate system. The point source of magnitude Q is assumed to be instantaneous and may be modeled as a Dirac delta function which is absorbed in an infinitesimal depth at the surface

$$g = Q \,\delta(x'' - \zeta') \,\delta(y'' - \eta') \,\delta(z'' - 0) \,\delta(\tau - 0) \tag{9}$$

Using the appropriate form of the multidimensional Green's function [cf. 24] with Eqs. (8) and (9), the temperature distribution in the body due to a point source is given by

$$T = \frac{Q}{\rho c} \frac{1}{abL} \left[ 1 + 2 \sum_{n=1}^{\infty} \exp\left(\frac{-n^2 \pi^2 a_x t}{a^2}\right) \cos\left(\frac{n\pi x}{a}\right) \cos\left(\frac{n\pi \zeta}{a}\right) \right]$$
$$\times \left[ 1 + 2 \sum_{n=1}^{\infty} \exp\left(\frac{-n^2 \pi^2 a_y t}{b^2}\right) \cos\left(\frac{n\pi y}{b}\right) \cos\left(\frac{n\pi \eta}{b}\right) \right]$$
$$\times \left[ 1 + 2 \sum_{n=1}^{\infty} \exp\left(\frac{-n^2 \pi^2 a_z t}{L^2}\right) \cos\left(\frac{n\pi z}{L}\right) \right]$$
(10)

where  $a_x$ ,  $a_y$ , and  $a_z$  are the thermal diffusivities in the x, y, and z directions, respectively. In order to determine the temperature distribution due to the finite area flash source (Fig. 1b), we may use superposition to include the contributions from a continuously distributed set of point sources by integrating Eq. (10) over the flash region.

$$T_s = \int_{\mathcal{A}} T(\zeta, \eta, t) \, d\zeta \, d\eta \tag{11}$$

Thus, Eq. (11) presents a general solution of the temperature response due to an arbitrary symmetric flash geometry. In the case of a square planar source of width and height  $2\Delta$  (Fig. 1), this response becomes

$$T_{s} = \frac{Q}{\rho c} \frac{1}{abL} \left[ \varDelta + 2 \sum_{n=1}^{\infty} \exp\left(\frac{-n^{2}\pi^{2}a_{x}t}{a^{2}}\right) \cos\left(\frac{n\pi x}{a}\right) \frac{a}{n\pi} \sin\left(\frac{n\pi \varDelta}{a}\right) \right] \\ \times \left[ \varDelta + 2 \sum_{n=1}^{\infty} \exp\left(\frac{-n^{2}\pi^{2}a_{y}t}{b^{2}}\right) \cos\left(\frac{n\pi y}{b}\right) \frac{b}{n\pi} \sin\left(\frac{n\pi \varDelta}{b}\right) \right] \\ \times \left[ 1 + 2 \sum_{n=1}^{\infty} \exp\left(\frac{-n^{2}\pi^{2}a_{z}t}{L^{2}}\right) \cos\left(\frac{n\pi z}{L}\right) \right]$$
(12)

Equation (12) reduces to the one-dimensional Parker solution if the limits of integration in Eq. (11) are b and a. Other closed-form solutions may be obtained for simple finite waveforms and simple pulse geometries which may be described with linear functions. A simple uniform pulse of finite duration may be included in Eq. (10) by replacing t with  $t-t_c$  and integrating with respect to  $t_c$  over the pulse duration in Eq. (11). Without loss of generality, numerical integration involving superposition of solutions may be used to incorporate finite pulse time effects of arbitrary waveforms and pulses of arbitrary geometry. In such cases, it may be more feasible to use Eq. (8) directly without the use of Dirac delta functions. For the common pulse geometry of a circular spot of radius r, Eq. (11) becomes

$$T_{s} = \int_{0}^{r} \int_{0}^{\sqrt{r^{2} - \zeta^{2}}} T(\zeta, \eta, t) \, d\eta \, d\zeta$$
(13)

and for a line source of width 2A, Eq. (11) reduces to

$$T_{s} = \frac{Q}{\rho c} \frac{1}{bL} \left[ \Delta + 2 \sum_{n=1}^{\infty} \exp\left(\frac{-n^{2}\pi^{2}a_{y}t}{b^{2}}\right) \cos\left(\frac{n\pi y}{b}\right) \frac{b}{n\pi} \sin\left(\frac{n\pi\Delta}{b}\right) \right] \\ \times \left[ 1 + 2 \sum_{n=1}^{\infty} \exp\left(\frac{-n^{2}\pi^{2}a_{z}t}{L^{2}}\right) \cos\left(\frac{n\pi z}{L}\right) \right]$$
(14)

Upon inspection of Eq. (14), it is evident that as the ratio  $\alpha_z/L^2$  becomes infinite, the temperature solution approaches that of a one-dimensional heat conduction problem which is assumed in the measurement of thin-film conductivity by the flash method [17].

# 2.2. A Semiinfinite Solid

By considering the stolid in Fig. 1 to be infinite in the x-y plane, the boundary conditions in Eqs. (4) and (5) become

$$\frac{\partial T}{\partial x'} = 0, \qquad x' = 0; \qquad T(x) = 0, \qquad x' = \infty$$
 (15)

$$\frac{\partial T}{\partial y'} = 0, \qquad y' = 0; \qquad T(y) = 0, \qquad y' = \infty$$
(16)

Using the appropriate form of the Green's function along with Eqs. (8) and (9), the temperature distribution due to an instantaneous point source in the original frame is

$$T = \frac{Q}{\rho c} \frac{1}{L} \left[ (4\pi a_x t)^{-1/2} \left( \exp\left(-\frac{(x-\zeta)^2}{4a_x t}\right) + \exp\left(-\frac{(x+\zeta)^2}{4a_x t}\right) \right) \right]$$
$$\times \left[ (4\pi a_y t)^{-1/2} \left( \exp\left(-\frac{(y-\eta)^2}{4a_y t}\right) + \exp\left(-\frac{(y+\eta)^2}{4a_y t}\right) \right) \right]$$
$$\times \left[ 1 + 2\sum_{n=1}^{\infty} \exp\left(\frac{-n^2 \pi^2 a_z t}{L^2}\right) \cos\left(\frac{n\pi z}{L}\right) \right]$$
(17)

Through the superposition principle presented by Eq. (11), any arbitrary symmetric flash geometry can be handled. Assuming a square planar geometry of width and height  $2\Delta$ , the temperature distribution becomes

$$T_{s} = \frac{Q}{\rho c} \frac{1}{L} \left[ \frac{1}{2} \operatorname{Erf} \left( \frac{\Delta - x}{2(a_{x}t)^{1/2}} \right) + \frac{1}{2} \operatorname{Erf} \left( \frac{\Delta + x}{2(a_{x}t)^{1/2}} \right) \right] \\ \times \left[ \frac{1}{2} \operatorname{Erf} \left( \frac{\Delta - y}{2(a_{y}t)^{1/2}} \right) + \frac{1}{2} \operatorname{Erf} \left( \frac{\Delta + y}{2(a_{y}t)^{1/2}} \right) \right] \\ \times \left[ 1 + 2 \sum_{n=1}^{\infty} \exp\left( \frac{-n^{2}\pi^{2}a_{z}t}{L^{2}} \right) \cos\left( \frac{n\pi z}{L} \right) \right]$$
(18)

whereas a line source of width  $2\Delta$  oriented in the x direction is given by

$$T_{s} = \frac{Q}{\rho c} \frac{1}{L} \left[ \frac{1}{2} \operatorname{Erf} \left( \frac{\Delta - y}{2(a_{y}t)^{1/2}} \right) + \frac{1}{2} \operatorname{Erf} \left( \frac{\Delta + y}{2(a_{y}t)^{1/2}} \right) \right] \\ \times \left[ 1 + 2 \sum_{n=1}^{\infty} \exp \left( \frac{-n^{2}\pi^{2}a_{z}t}{L^{2}} \right) \cos \left( \frac{n\pi z}{L} \right) \right]$$
(19)

In applying Eq. (13) to Eq. (17), the result converges along principal directions to the solution of a circular disk source on a transversely isotropic semiinfinite plane given by Carslaw and Jaeger [25]. Such an approach is typical in the radial flash method and, therefore, shows the agreement of the two approaches. The results presented thus far provide the temperature distribution for an orthotropic adiabatic body subjected to an instantaneous pulse. These results may be extended to account for linear heat losses (e.g., convection) with simple modifications [26]. For this analysis, the simple adiabatic case presented is only considered.

# 3. DETERMINATION OF THERMAL DIFFUSIVITY IN MULTIPLE DIRECTIONS

The thermal diffusivity of materials may be determined from temperature-time history data obtained from experiments similar to the radial flash method By measuring the temperature response at multiple locations simultaneously, it is possible to measure the thermal diffusivity in both directions parallel and perpendicular to the flash in one experiment. This may be achieved through monitoring the front and rear surfaces simultaneously or by single surface monitoring. Monitoring a single surface alone yields the two principal diffusivities perpendicular to the flash direction in a straightforward manner. Once the in-plane parameters are determined, the principal diffusivity parallel to the flash direction may be found. This is facilitated by the correlation of the temperature response at a point with the full analytical temperature solution since two of the material parameters are known. In the situation where both the front and the rear surfaces are monitored, the through-thickness thermal diffusivity can be determined from the ratio of the front and rear temperatures of any two points along the same axis [12]. For two points along the axis of the flash [e.g., T(0, 0, 0, t) and T(0, 0, L, t)], this temperature ratio will vary as

$$\frac{T_1}{T_0} = \frac{1+2\sum_{n=1}^{\infty} (-1)^n \exp\left(-\frac{n^2 \pi^2 a_z t}{L^2}\right)}{1+2\sum_{n=1}^{\infty} \exp\left(-\frac{n^2 \pi^2 a_z t}{L^2}\right)}$$
(20)

Equation (20) is the characteristic equation which describes the temperature ratio of any two points along the z axis from the front and rear surfaces. Equation (20) is independent of the heat absorbed and is valid regardless of the lateral geometry of the sample, whether heat flow is unidirectional or multidirectional, and regardless of the geometry of the incident flash. This is due to the fact that any dependence which the temperature response may have on the other coordinate directions is canceled out when forming this ratio. Thus, linear heat losses in lateral directions do not affect results obtained according to Eq. (20). Corrections for heat losses from the front and rear surfaces may be made using any number of the aforementioned techniques applied to the Parker flash method.

In order to determine the in-plane thermal diffusivities, a procedure similar to that used to develop Eq. (20) is employed. A ratio of any two temperatures recorded on the front or rear surface along a principal axis will also yield the thermal diffusivity in that particular coordinate direction. The semianalytical approach leads to the simplest data reduction methods along principal axes. This occurs since the foundation of the approach, Eq. (1), is written in the principal coordinate system. Under certain conditions, the analysis is not limited to points which lie only along the principal axes. This involves conditions where one principal axis is aligned perpendicular to the plane on which the flash is incident. With the use of circular flash geometries in such cases, knowledge of the principal directions within the plane need not be known a priori. In such cases, multiple temperature ratios with respect to the center of the flash may be taken at fixed angular distances, as shown in Fig. 2. In the analysis of each of these temperature ratio responses, it can be assumed that a coupling exists between the two in-plane principal diffusivities. In addition, it is assumed that the



Fig. 2. Depiction of experimental coordinate frame with arbitrary rotation relative to the principal axes.

experimental axes are rotated relative to the principal axes by an angle  $\theta_1$ . By rewriting the analytical temperature solution in terms of the unknown principal coordinate directions [i.e.,  $X = X' \cos(\theta_n) + Y' \sin(\theta_n)$ ,  $Y = X' \sin(\theta_n) + Y' \cos(\theta_n)$ ], we introduce an additional fitting parameter into our model. An iterative procedure can be used to determine both the angle  $\theta_1$  and the two principal diffusivities until the solutions converge for all in-plane temperature ratios. It is assumed in this case that  $\theta_2$  is the known fixed angular distance. Since the angle  $\theta_1$  is also determined using this procedure, the principal direction of heat flow may also be deduced. The use of several temperature ratios from fixed angular distances to determine principal diffusivities and directions of heat flow is analogous to the use of the strain gauge rosette in solid mechanics for the determination of principal stresses and strains.

Since a coupling does not exist along principal axes, the determination of thermal diffusivity is more straightforward under these conditions. For a body subjected to a line or square flash source, the thermal diffusivity in the x direction may be found from the ratio of the temperatures at two collinear points. Assuming  $T_1$  is at the origin and  $T_2$  is at some position x = X, the ratio of the temperatures taken at these two points will vary according to

$$\frac{T_2}{T_1} = \frac{\Delta + 2\sum_{n=1}^{\infty} \exp\left(-\frac{n^2 \pi^2 a_x t}{a^2}\right) \cos\left(\frac{n\pi X}{a}\right) \frac{a}{n\pi} \sin\left(\frac{n\pi \Delta}{a}\right)}{\Delta + 2\sum_{n=1}^{\infty} \exp\left(-\frac{n^2 \pi^2 a_x t}{a^2}\right) \frac{a}{n\pi} \sin\left(\frac{n\pi \Delta}{a}\right)}$$
(21)

for a bounded region or the following for a semiinfinite solid:

$$\frac{T_2}{T_1} = \frac{\left[\operatorname{Erf}\left(\frac{\Delta - X}{2(a_x t)^{1/2}}\right) + \operatorname{Erf}\left(\frac{\Delta + X}{2(a_x t)^{1/2}}\right)\right]}{\left[2\operatorname{Erf}\left(\frac{\Delta}{2(a_x t)^{1/2}}\right)\right]}$$
(22)

It should be noted that Eqs. (21) and (22) are independent of the y and z coordinates. Therefore, these are the characteristic equations which describe the temperature ratio due to an instantaneous flash of any two points along the x axis in the same plane provided that it is a principal axis for heat conduction. These equations hold regardless of unidirectional or multidirectional heat flow, and the presence of heat losses in the other coordinate directions. The independence of this result to heat losses along

one of the lateral boundaries was shown experimentally and numerically in Ref. 26. Such independence is not a characteristic of the radial flash method. In addition, these equations hold for both isotropic and orthotropic materials. Thus, the response of the temperature ratio along a principal axis may be characterized by a solution based on isotropic material properties. Hence, it is understood that the radial flash technique developed by Donaldson and Taylor [12] may be applied to orthotropic materials provided the measurements are taken along the principal axes. However, the present derivations provide a more robust procedure in that it predicts the temperature distribution within the solid for any flash geometry and duration and may be used for off-axis measurements where a coupling of principal diffusivities exists. This result allows for noncircular sample geometries and measurements on orthotropic materials where the principal directions of heat flow are unknown. In addition, this technique provides a procedure to map diffusivity changes in materials with evolving microstructures which may induce changes in principal directions. Indeed, the present solution can be used to assess whether orthotropy of heat conduction actually exists for materials which are assumed to be orthotropic.

For samples which cannot be considered semiinfinite with respect to heat flow, the lateral boundary conditions of the specimen will play an important role in determining the temperature distribution in the solid. Modifications to the temperature solutions may be performed as aforementioned using heat loss correction procedures. However, if the temperature measurement is taken over a sufficiently small time, the heat flow will not interact with the boundaries, and thus, no heat loss corrections will be needed. Such procedures are viable for materials possessing small  $a/L^2$ ratios. Due to the independence of thickness displayed in Eqs. (21) and (22), this measurement technique may be used on samples of any thickness, providing a significant advantage for thin films which display statistical homogeneity. Films which are on the order of microns in thickness do not always display homogeneous behavior and, therefore, may not be amenable to this technique. Thick section samples coupled with rear surface imaging also limit the applicability of this method. In such cases, front surface imaging should be used to determine the principal diffusivities.

As stated previously, one of the problems associated with determining the in-plane diffusivity is the loss of accuracy compared to the unidirectional flash method. This is due to the sensitivity of the estimated parameter on the accuracy of the distance between the two temperatures recorded in the in-plane direction. Errors associated with the precise measurement of this distance may be eliminated by introducing it as a second variable in any parameter estimation procedure. A plot of the sensitivity coefficients of the thermal diffusivity and distance parameters for



Fig. 3. Temperature ratio and sensitivity coefficients for the thermal diffusivity and position of the second temperature measurement for Eq. (21). ( $\bigcirc$ ) Temperature ratio; ( $\triangle$ ) position coefficient; ( $\cdots$ -) diffusivity coefficient.

Eq. (21) is shown in Fig. 3. In analyzing this plot, it can be seen that the sensitivity of the position of the second temperature point and the thermal diffusivity are independent parameters [27]. Thus, it is possible to use a least-squares fitting procedure with these two parameters. These parameters can be determined independently, increasing the accuracy of in-plane diffusivity methods. By taking the ratio of the sensitivity coefficients of these two parameters will become difficult or impossible for Fourier numbers greater than 0.01. To verify this procedure, a temperature-time data set with known parameters was created. Using a nonlinear least-squares fitting procedure and random initial guesses for the parameters, the values were determined for both variables.



Fig. 4. Range of independence for the two-parameter estimation based on the slope of the sensitivity coefficient ratio. ( $\longrightarrow$ ) Diffusivity/position coefficient ratio; ( $\triangle$ ) temperature ratio.

In order to investigate further the validity of the derived temperature solutions, a finite-element study was performed. A three-dimensional rectangular geometry was analyzed using the commercial finite-element code ABAQUS [28]. The flash was input into the model as an approximation of an instantaneous step function of a uniform flux. This flux was incident over a finite width of several elements for a duration of 1 ms. The half-flash width,  $\Delta$ , was 0.45 cm, while the width, height, and thickness were 7.56, 7.56, and 2.0 cm, respectively. The thermal conductivity ratios were  $K_x/K_x = 5$ and  $K_z/K_y = 10$ , and the heat capacity was  $1 \text{ J} \cdot \text{cm}^{-3} \cdot \text{K}^{-1}$ . Temperature ratios of the nodes along the axis of the flash on the front and rear surfaces were calculated and correlated using Eq. (20). For the in-plane simulations, temperature ratios of in-plane nodes on the rear surface with respect to the node at the center of the flash (x = y = 0, z = 2.0) were calculated for correlation with Eq. (21). For the in-plane directions, two temperature ratios were formed for each direction with respect to the center temperature. The locations of the second temperature points were 0.49 and 0.82 cm away from the center along both the x and the y axes. This was done to confirm the results and convergence of the model at multiple locations along the same direction. Using a least-squares routine employing Eqs. (20) and (21) to fit the data, the calculated thermal diffusivity was within 0.2% of the known values for all simulations. Plots of the data correlation are shown in Figs. 5 and 6. In Fig. 6, only one curve is shown for both in-plane directions. Since the geometric model and temperature measurement points are symmetric, the responses in the x and y directions collapse onto the same nondimensional curve. Correlations with front surface simulations (z=0) using Eqs. (21) and (22) showed similar results. This behavior shows the depth independence of the in-plane method and



Fig. 5. Comparison of numerical and semianalytical results using Eq. (20) for through-thickness diffusivity. (—) Semianalytical; ( $\bigcirc$ ) ABAQUS [28].



**Fig. 6.** Comparison of numerical and semianalytical results using Eq. (21) for in-plane thermal diffusivity determination in the x and y directions. N = normalized position coordinate x/a or y/b. In the case shown, a = b = 7.56 cm. (--) Semi-analytical; ( $\bigcirc$ ) ABAQUS [28]; ( $\square$ ) ABAQUS [28].

that the sample behaved as a semiinfinite medium over the duration of the numerical analysis.

# 4. EXPERIMENTS

In order to study the feasibility of implementing this technique, an experimental program was undertaken. Room-temperature flash diffusivity measurements were made at Oak Ridge National Laboratory on carbon-coated Nicalon-reinforced lithium aluminosilicate glass (CC-SiC/LAS). Details of the experimental procedure, as well as results on a second material system, are given in Ref. 26. The samples were made with unidirectional reinforcement and contained 40% fiber volume fraction. All tests were performed on a single panel which was  $15.24 \times 15.24 \times 0.251$  cm thick. For comparison, small samples were also cut in order to measure the thermal diffusivity along and transverse to the fiber direction with the Parker technique. These samples were 0.278 cm long, 1.27 cm wide, and 0.251 cm thick. Samples were checked with an optical microscope to ensure that no damage was introduced from cutting, which may affect the measurements.

The panel was irradiated on the plane transverse to the fibers by a xenon flash lamp. A mask with a square hole  $1.27 \times 1.27$  cm was used to provide the desired flash geometry. Care was also taken to reduce the amount of light dispersion passing through the mask by mounting the panel 0.5 mm from the mask. The temperature on the rear surface was monitored using a  $256 \times 256$  focal plane array InSb infrared detector with a temperature resolution of  $0.015^{\circ}$ C. The rear surface temperature was recorded in snapshot mode at a rate of 10 Hz for a period of 20 s.

The in-plane temperature ratios were formed by using the temperature at the center of the flash and at a point between 0.6 and 0.75 cm away. Measurements were made both along the fiber and transverse to the directions. A nonlinear least-squares procedure was used to fit the experimental data and to determine both the thermal diffusivity and the distance between the two in-plane temperatures (Fig. 7). The results of this procedure were compared to the Parker-type flash method with heat loss correction taken into account by the method of Clark and Taylor [7]. The results, presented in Table I, show excellent agreement between the two methods, with thermal diffusivity values of 0.008344 and 0.00796 cm<sup>2</sup> · s<sup>-1</sup> in the fiber and transverse directions, respectively. These values are consistent with the results presented for this material system [29]. Thus, it is seen that the in-plane method combined with the two-parameter least-squares estimation is an



Fig. 7. Correlation of experimental data from CC-SiC/LAS using a two-parameter nonlinear least-squares procedure and Eq. (21). (A) Transverse direction ( $\alpha = 0.008 \text{ cm}^2 \cdot \text{s}^{-1}$ ). (B) Fiber direction ( $\alpha = 0.00836 \text{ cm}^2 \cdot \text{s}^{-1}$ ). (C) Experimental; (----) semianalytical.

Fiber direction	Diffusivity $(cm^2 \cdot s^{-1})$	Transverse direction	Diffusivity $(cm^2 \cdot s^{-1})$
Mean	0.00834	Mean	0.00796
SD	0.00018	SD	0.00011
Clark and Taylor [7]	0.0085	Clark and Taylor [7]	0.0078
% difference	1.9	% difference	2.0

Table I. Comparison of In-Plane and Unidirectional Flash Techniques

accurate nondestructive method. It should also be noted that the data reduction scheme used did not account for heat loss correction for the inplane measurements. Due to the very low ratio of  $\alpha$  to the in-plane dimensions ( $\approx 0.001$ ), there was no heat loss at the lateral boundaries of the sample for the time frame of the in-plane tests.

### 5. CONCLUSION

The generalization of the radial flash technique presented here can be used as a powerful tool in the determination of thermal diffusivity. The analytical formulation provides a general approach to the application of flash diffusivity to anisotropic materials which is valid for an arbitrary flash geometry and flash duration. It is not limited to measurements along principal axes of heat flow. Thermal diffusivities can be inferred from experiments by fitting and matching a semianalytical scheme where the diffusivities are determined parameters. By including a second parameter into the nonlinear least-squares fitting procedure, the accuracy of the method is shown to be comparable to the widely used Parker flash-type method. For the measurement of in-plane properties, the use of materials and geometries which may be considered semiinfinite over the test duration require no heat loss correction schemes. The further development of this technique, coupled with micromechanical studies, may also lead to a rapid quality control measurement system, especially for components designed for thermal applications. Since no destructive sectioning is required, the application of this procedure should benefit the measurement of thermal diffusivity and conductivity of expensive and unique engineering components, and allow for the in situ characterization of materials. Furthermore, progressive degradation of thermal diffusivity and conductivity of orthotropic solids and thins films due to process or service-induced damage may potentially be resolved with this technique in a nonintrusive manner. Connections between material damage or degradation and conductivity may therefore permit exploitation of the technique as an indirect measure of such degradation.

# ACKNOWLEDGMENT

The research was sponsored by the Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Transportation Technologies, as part of the High Temperature Materials Laboratory User Program, Oak Ridge National Laboratory, managed by Lockheed Martin Energy Research Corporation for the U.S. Department of Energy under Contract DE-AC05-960R22464.

# REFERENCES

- 1. G. Danielson and P. Sidles, *Thermal Conductivity 2*, R. P. Tye, ed. (Academic, New York, 1969), pp. 149–201.
- 2. Y. S. Touloukian, R. W. Powell, C. Y. Ho, and M. C. Nicolaou, *Thermophysical Properties of Matter, Vol. 10. Thermal Diffusivity* (1F1/Plenum Press, New York, 1973), p. 82.
- 3. W. J. Parker, R. J. Jenkins, C. P. Butler, and G. L. Abbott, J. Appl. Phys. 32:1679 (1961).
- 4. J. A. Cape and G. W. Lehman, J. Appl. Phys. 34:1909 (1963).
- 5. R. D. Cowan, J. Appl. Phys. 34:926 (1963).
- 6. R. C. Heckman, J. Appl. Phys. 44:1455 (1973).
- 7. L. M. Clark and R. E. Taylor, J. Appl. Phys. 46:714 (1975).
- 8. A. Degiovanni, Rev. Gen. Therm. 16:417 (1977).
- 9. L. A. Gritzo and E. Anderson, *Thermal Conductivity 22*, T. W. Tong, ed. (Technomic, Lancaster, PA, 1992), pp. 59-69.
- 10. A. B. Donaldson, J. Frank. Inst. 294:275 (1972).
- 11. A. B. Donaldson, J. Appl. Phys. 43:4226 (1972).
- 12. A. B. Donaldson and R. E. Taylor, J. Appl. Phys. 46:4584 (1975).
- 13. F. I. Chu, R. E. Taylor, and A. B. Donaldson, J. Appl. Phys. 51:336 (1980).
- 14. M. Amazouz, C. Moyne, and A. Degiovanni, High Temp.-High Press. 19:37 (1987).
- 15. C. L. Choy, W. P. Leung, and Y. K. N, J. Polym. Sci. B 25:1779 (1987).
- 16. H. Ohta, H. Shibata, and Y. Waseda, Rev. Sci. Instrum. 60:317 (1989).
- 17. H. Shibata, H. Ohta, and Y. Waseda, Mater. Trans. JIM 32:837 (1991).
- 18. S. D. Preston, High Temp.-High Press. 27/28:111 (1995/1996).
- J. D. Lin and L. J. Sheu, *Thermal Conductivity* 22, T. W. Tong, ed. (Technomic, Lancaster, PA, 1992), pp. 634–645.
- 20. H. J. Lee and R. E. Taylor, J. Appl. Phys. 47:148 (1976).
- 21. H. J. Lee and R. E. Taylor, J. Heat Trans. 100:720 (1978).
- 22. M. S. Deshpande, R. H. Bogaard, and R. E. Taylor, Int. J. Thermophys. 2:357 (1981).
- 23. D. Balageas and A. Luc, AIAA J. 24:109 (1986).
- 24. M. N. Özisik, Heat Conduction (John Wiley, New York, 1993).
- 25. H. S. Carslaw and J. C. Jaeger, Conduction of Heat in Solids (Clarendon, Oxford, 1959).
- S. Graham, D. L. McDowell, R. B. Dinwiddie, *Thermal Conductivity 24*, P. S. Gaal, ed. (Technomic, Lancaster, PA, 1999), pp. 241–252.
- 27. J. V. Beck and K. J. Arnold, *Parameter Estimation in Engineering and Science* (John Wiley, New York, 1971), pp. 17–23.
- 28. Hibbitt, Karlsson, and Sorensen, Inc., ABAQUS, Version 5.7 (1997).
- 29. J. J. Brennan, L. D. Bentsen, and D. P. H. Hasselman, J. Mat. Sci. 17:2337 (1982).