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Modeling Flash Diffusivity Experiments in Two Dimensions for Thick Samples

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The laser flash method for measuring thermal diffusivity is well established and has been in use for many years. Early analysis methods employed a simple model, in which one-dimensional transient conduction was assumed, with insulated surfaces during the time subsequent to the flash. More recently, models of greater sophistication have been applied to flash diffusivity experiments using nonlinear regression. The advanced models have achieved highly accurate agreement with experimental data taken from thin samples, on the order of 1 mm in thickness. As samples become thicker, models that neglect edge losses can lose some conformity to the experimental data. The present research involves the application of a two-dimensional model, which allows for penetration of the laser flash into the sample. The accommodation of the flash penetration is important for porous materials, where the coarseness of the porosity is more than 1% of the sample thickness. Variability of the area of incidence of the flash is also investigated to determine the effect on the model and the results. Statistical methods are used in order to make a determination as to the validity of the two-dimensional model, as compared with the one-dimensional analysis method.

Nomenclature

a	=	depth of flash penetration, m
Bi	=	Biot number, hL/k
c	=	specific heat, J/kg-K
h	=	convection coefficient, W/m^2-K
k	=	thermal conductivity, W/m-K
L	=	thickness of sample, m
q_o	=	magnitude of heat pulse, J/m^2
R	=	sum of squares of residuals, K^2
r	=	radial coordinate of sample, m
ρ	=	density, kg/m^3
r_h	=	radius of heating, m
r_o	=	outer radius of sample, m
s	=	mean sum of squares of residuals, K^2
$T(r,x,t)$	=	temperature in sample above ambient, °C
x	=	axial coordinate of sample, m
α	=	thermal diffusivity, m^2/s

Introduction

THE method of flash heating for the purpose of measuring thermal diffusivity has been in use for over 40 years. Consequently, there are several companies that market the laboratory equipment needed to make convenient and systematic measurements using this method. The method carries the advantages of being rapid in terms of execution because the samples are small, causing transient heat conduction to take place quickly. The samples are typically disc-shaped and are the size of a coin, being 1 or 2 cm in diameter and approximately 1 mm in thickness. In some instruments, multiple samples can be tested sequentially, because they are loaded into a multichamber carousel, much like the chambers in a revolver.

The flash heating of the samples typically takes place over a very short duration of time, on the order of several milliseconds. The magnitude of the flash is usually quite intense, on the order of several kilowatts per square millimeter. Subsequent to the flash, the temperature begins to rise on the opposite side of the sample as conduction takes place. This thermal transient is recorded with an optical temperature measurement system, which is usually capable of sampling rates on the order of tens of kilohertz. The noncontact temperature measurement method allows experiments to be performed at very high temperatures. Once the temperature history is obtained, it is analyzed in order to determine the thermal diffusivity of the sample. The laser flash system employed in this work, which was manufactured by Anter Corporation, used a neodymium glass laser with a wavelength of 1.053 μ.

The method of analysis used in estimating thermal diffusivity in the flash experiments can have a significant impact on the results. The first experiments performed using the flash method ignored heat losses from the sample. Once the energy from the flash was absorbed into the material, it was assumed to remain in the material for the duration of the experiment. Indeed, because the experiments are normally performed in a vacuum, the heat losses are minimal over the time window during which the conduction transient takes place in a highly conductive thin sample. This is particularly true at moderate ambient temperatures. In the analysis used by Parker et al. [1], only the amount of time required for the sample to reach half of its ultimate temperature rise was ultimately used in computing the thermal diffusivity of the sample. Cowan [2] modified the analysis method by the use of correction factors to account for heat losses. Charts were developed in this case to provide analysts with heat loss correction factors appropriate for various heat loss conditions. This work was expanded by Clark and Taylor [3] to include correction factors for the outer edge of the samples, providing some accommodation of two-dimensional effects. Further refinement in the analysis of flash diffusivity experiments was provided by Koski [4] as he fitted mathematical models to the experimental data by using least squares. Because this method used the entire time domain of the transient, instead of relying on only one data point in calculating thermal diffusivity, the results were much less susceptible to measurement errors near the point of half the maximum temperature rise. This work was further expanded to include heat losses by Taylor [5] as well as various shapes of the laser pulse. A summary of some of these methods is made by Raynaud et al. [6], where a comparison is made of the adequacy of these models. Likewise, similar comparisons are made by Beck and Dinwiddie [7], particularly evaluating the necessity for making distinctions in the convection coefficients used for opposite sides of the sample. McMasters et al. [8] discusses a method for determining the thermal conductivity of a thin film on a known substrate using the flash diffusivity method. For this analysis, the volumetric heat capacity of the film and the substrate must both be known. Research that is similar to the present work, although not involving flash diffusivity testing, is described by Koo et al. [9] and

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Hender [10]. In both of these cases, adjustments were made in parameters in mathematical models to more adequately allow the models to conform to experimental measurements.

All of the previously mentioned methods that involve flash diffusivity experiments assumed that the laser flash heated only the surface of the sample at the instant of the flash. The concept of the penetrating effect of the flash heating was first addressed by McMasters et al. [11], examining porous surfaces of carbon bonded carbon fiber materials. The residuals from this analysis method, which are simply the difference between the mathematical model and the experimental measurements at each time step, were found to be greatly reduced when compared with the residuals from models accounting for surface heating only. Most importantly, a characteristic signature in the residuals was found to be greatly reduced by accounting for flash penetration.

The present research examines an analysis method that makes use of a fully two-dimensional numerical direct model, fitted to the experimental measurements by nonlinear regression. This model not only accommodates edge heat losses, but also allows for partial surface heating and flash penetration. Specifically, all one-dimensional models assume that the heating of the sample is uniform over the entire surface. By contrast, the present research, taking advantage of the fully two-dimensional model, accounts for uneven heating of the sample where the flash may be somewhat concentrated in the center of the heated surface. A significant reduction of the standard deviation of the residuals was achieved using this method. Moreover, the statistical significance of the additional parameter associated with nonuniform surface heating was demonstrated by using the "F test," and a noticeable difference in the estimated thermal diffusivity was found. A one-dimensional least-squares model was also used for evaluation of the data as a basis of comparison.

Description of Experiment

The material tested for this research was Alton foam, and the experiments were performed at the High Temperature Materials Laboratory, a division of the Oak Ridge National Laboratory, in Oak Ridge, Tennessee. The Alton foam material is a rigid, abrasive, porous, carbon-based substance. It is typically used for insulation applications in very-high-temperature environments. When examined on a microscopic scale, the material has quite an irregular surface, even though it is squarely cut, primarily because of the porosity of the material. The scale of the voids in the material, however, is at least two orders of magnitude smaller than the overall dimensions of the sample, allowing for continuum modeling to hold validity. Although the determination of the point at which a continuum model loses its validity is beyond the scope of this research, the improvement shown in the results obtained by this and other analysis methods demonstrates the validity of the continuum model for the samples chosen. The porous property of the material allows the laser flash to heat the material at a deeper level than would occur in heating a nonporous material with a smooth surface. Accounting for this flash penetration in the direct solution of the parameter estimation method can produce different results than those generated using models that do not account for penetration, as described in [11]. The sample thickness was 2.280 mm, more than twice the thickness of many of the samples tested using flash diffusivity instruments. One reason for the larger thickness required in these samples is the difficulty in cutting the material to the desired sample size without having the sample break, due to the porosity and brittleness of the material.

As noted in the introduction, heating is normally considered uniform on the incident surface of the sample in flash diffusivity tests. The model used in this research accounts for the possibility that the heating on the surface is nonuniform. Figure 1 shows a diagram of the sample used in this research, showing the heated area of the sample. The heating in this model is considered uniform over the heated area shown, but the size of the heated area can be treated as a parameter, if desired, using one of the analysis models developed as part of this research.

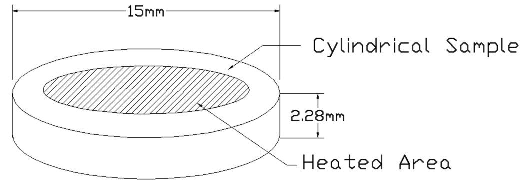

Fig. 1 Diagram of the sample showing the area of flash heating.

Analysis Method

In contrast to the analysis methods used in [1–4], the present method provides a full-duration temperature calculation, fitted to the experimental data using least squares over the entire transient time domain of the experiment. The principles of [12] were used in accomplishing the least-squares fit, including the calculation of sensitivity coefficients for each of the parameters over the entire time domain. These sensitivity coefficients were then employed with a Newton–Raphson routine to iteratively adjust the parameters to arrive at a fit producing a minimum sum of squares of errors. The convergence criteria used in this work required that the change in all parameters be less than 0.1% between iterations. The direct solution was a two-dimensional cylindrical-geometry configuration solved by a numerical scheme to find $T(r, x, t)$ of the transient conduction equation, specifically

$$\frac{1}{\alpha}\frac{\partial T}{\partial t} = \frac{1}{r}\frac{\partial}{\partial r}\left(r\frac{\partial T}{\partial r}\right) + \frac{\partial^2 T}{\partial x^2} \tag{1}$$

where α is thermal diffusivity (m^2/s). The boundary conditions were

$$k\frac{\partial T}{\partial x}\bigg|_{x=0} = h(T(r, 0, t) - T_\infty) \tag{2}$$

$$-k\frac{\partial T}{\partial x}\bigg|_{x=L} = h(T(r, L, t) - T_\infty) \tag{3}$$

$$-k\frac{\partial T}{\partial r}\bigg|_{r=r_o} = h(T(r_o, x, t) - T_\infty) \tag{4}$$

In these equations, h is the convection coefficient (W/m^2-K), k is thermal conductivity (W/m-K), T_∞ is the ambient temperature (K), and r_o is the outer radius of the sample.

Five models were applied to the experimental data individually and evaluated in terms of their adequacy. The five models used in this comparison were

model a: one-dimensional conduction assuming surface heating only

model b: dimensional conduction assuming surface heating only

model c: one-dimensional conduction assuming penetration of the flash

model d: two-dimensional conduction assuming penetration of the flash

model e: two-dimensional conduction assuming penetration of the flash and a nonuniform distribution of the flash over the face of the sample (i.e., $r_h < r_o$).

For the one-dimensional models, the radial derivatives are zero in Eq. (1) and the boundary condition given by Eq. (4) is not applicable. It is important to note that, for all of the two-dimensional models used, the boundary conditions and the differential equation being solved are the same. The only differences between the various models are in the initial conditions. The initial conditions for model e are

$$T(r > r_h, x, 0) = T_\infty \qquad T(r \le r_h, x, 0) = \frac{q_o}{\rho c}e^{-x/a} + T_\infty \tag{5}$$

where r_h is the radius (m) of the sample surface that is heated, a is the mean free path (m) of a photon in the material, ρ is density (kg/m^3), c

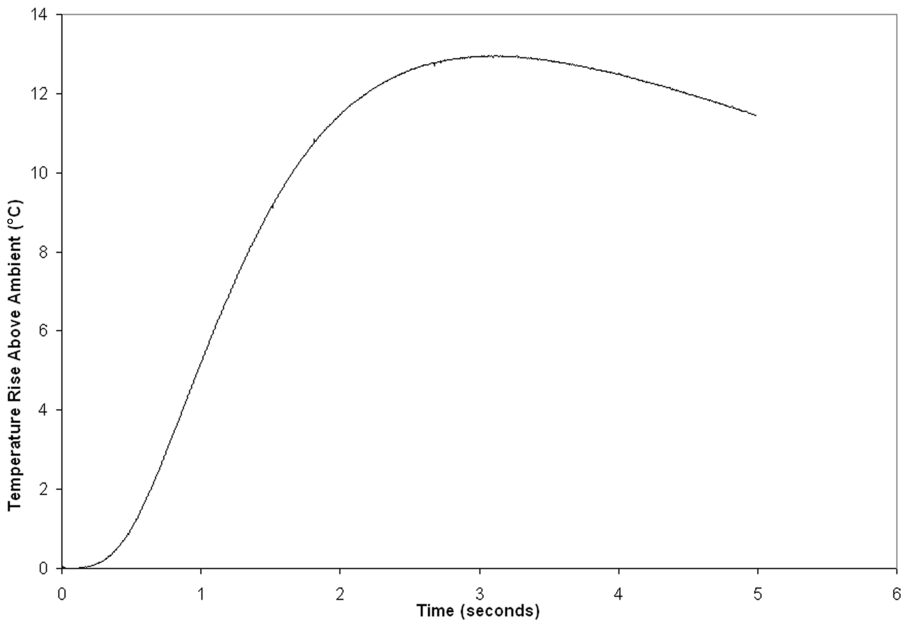

Fig. 2 Raw data from an experiment, showing the temperature as a function of time. The experimental curve and theoretical curve, using model e, are nearly indistinguishable at this scale.

is specific heat (J/kg K), and q_o is the amount of heat absorbed (J/m^2) from the flash. The parameter q_o is required to be calculated in order to establish the initial condition but is not desired as part of the objective of the experiment. It is, therefore, considered a "throwaway parameter." This parameter is used in the expression

$$\frac{q_o}{\rho c} e^{-x/a} \tag{6}$$

as part of Eq. (5). As such, Eq. (6) represents the temperature rise in the sample near the surface due to the flash. This is in accordance with Beer's law regarding attenuation of radiation in semitransparent substances. Likewise, thermal conductivity cannot be determined as part of the flash diffusivity experiments. It is lumped into the dimensionless Biot number (hL/k) and thermal diffusivity $(k/\rho c)$. The Biot number is considered as another unneeded parameter, along with q_o, but must be calculated in order to determine thermal diffusivity.

The initial conditions for model b are the same as those prescribed previously for model e, except $r_h = r_o$. Additionally, the parameter a, the flash penetration distance, is effectively zero, making the exponential expression irrelevant. For model d, $r_h = r_o$ but the a term is nonzero, making the exponential operative. As with the basic one-dimensional model a the most basic two-dimensional model b must compute three parameters (α, Bi, q_o). This model accommodates two-dimensional heat transfer with uniform heating across the entire irradiated surface of the sample and no flash penetration. If the model is to accommodate penetration of the flash beyond the sample surface, the parameter a must be used as a measure of the extent of the penetration, which is the case in models c and d. Likewise, if accommodation is to be made for incomplete heating over the sample surface, the parameter r_h is estimated as well, serving as a measure of the portion of the surface that is heated. This is dealt with by model e.

The numerical method used consists of a 100 node finite control volume grid, with 10 nodes along the radius and 10 nodes in thickness. A fully implicit solution method was used for stability, and the time steps were chosen corresponding to dimensionless times of 0.1 based on the smallest node spacing.

Results

Altogether, six experiments were conducted on the Alton foam samples. Two samples were analyzed and three experiments were performed on each sample. All six of these experiments were analyzed, and the results all exhibited the same trends. The results from only one of these experiments are presented here in order to minimize redundancy. Figure 2 shows the raw data from the experiment, with the peak temperature occurring in the vicinity of the 3 s mark, with the entire experiment lasting 5 s. At the scale used in Fig. 2, the theoretical and experimental curves, using model e, can be seen to line up essentially on top of one another and are not discernible from one another. Therefore, the residual plots of subsequent figures are used for the purpose of closer examination of the errors between calculated and experimental temperatures.

A summary of the results from each of these analysis methods is given in Table 1. The primary means of comparing the adequacy of the models is in the standard deviation of the residuals. The residuals are the individual differences between the measured data points and the calculated data points for each of the measurements in the experiment. As this standard deviation of the residuals becomes smaller, the model conforms more closely to the experimental data. The maximum temperature rise in this experiment was approximately 12 deg above ambient, and so the magnitude of the residuals shown in Table 1 are relatively small. However, there is a noticeable improvement in the conformance of the mathematical model to the laboratory measurements when the two-dimensional model is used. The improvement in conformance can be seen

Table 1 A comparison of the results of the five analysis models on the experiment

Analysis model	Diffusivity, $m^2/s \times 10^6$	Penetration, mm	Diameter, mm	Standard deviation of residuals, °C
a) 1-D flash	0.5451	na	na	0.110899
b) 2-D flash	0.4954	na	na	0.082413
c) 1-D flash	0.4684	0.2168	na	0.036103
d) 2-D flash	0.4307	0.1910	na	0.022246
e) 2-D flash	0.4485	0.1953	10.85	0.020813

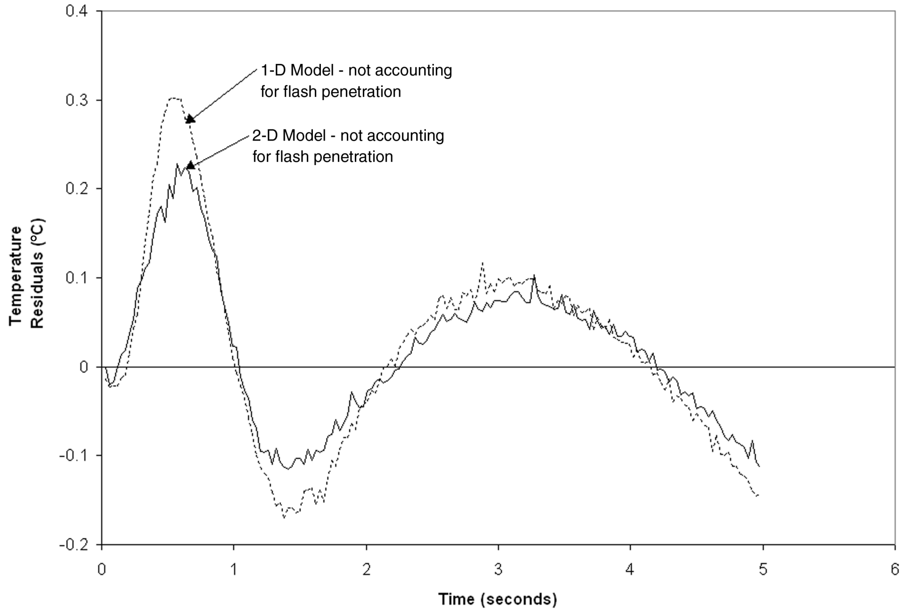

Fig. 3 Comparison of 1-D and 2-D models, which account for surface heating only (models a and c, respectively).

visually in examining a plot of the residuals as a function of time over the course of the experiment. If the model does not conform well to the experimental data, a characteristic signature will appear in the plot of the residuals. Figure 3 compares the first two models used in analyzing the data. Both of these models (one-dimensional and two-dimensional) make no allowance for penetration of the flash or for uneven heating. As can be seen in this figure, the two-dimensional model provides improved conformance to the experimental data. The signature in the residuals is reduced as well as the standard deviation of the residuals by nearly a factor of 4, as shown in Table 1. The peak value in this curve is smaller than the peak value in Fig. 2 by approximately a factor of 40. It can also be seen that there is a large peak just before a time of 1 s in this curve. This characteristic signature comes about because the model is not accounting for the premature arrival of the "wave" of heat diffusing through the sample recorded in the experiment. This is because the heating is assumed to be taking place only on the surface, and the effect of flash penetration is not being accounted for.

Figure 4 provides a visual comparison of the performance of the other three models used in the analysis of the experiment. Although the signatures associated with the results from these models appear similar to those of the first two models in Fig. 3, it should be noted that the temperature scale of Fig. 4 is much smaller than that of Fig. 3. Because the peak residual value shown in Fig. 3 is approximately 0.3, the peak of 0.06 in Fig. 4 represents approximately a five-fold reduction in the residual signature. As can also be seen in Fig. 4, both of the two-dimensional models featured outperform the one-dimensional model, even when all of them account for penetration of the laser flash. Moreover, the model that accounts for nonuniform heating over the surface, model e, provides the best performance of all five models. In this figure, a dip can be seen in the very early time steps. This reflects the fact that temperature recordings often read below ambient in the early stages of the experiment. Although this has no physical relevance to the actual temperature of the sample, it could be due to voltage fluctuations in the system at the time of the flash. An additional explanation for nonzero initial temperature

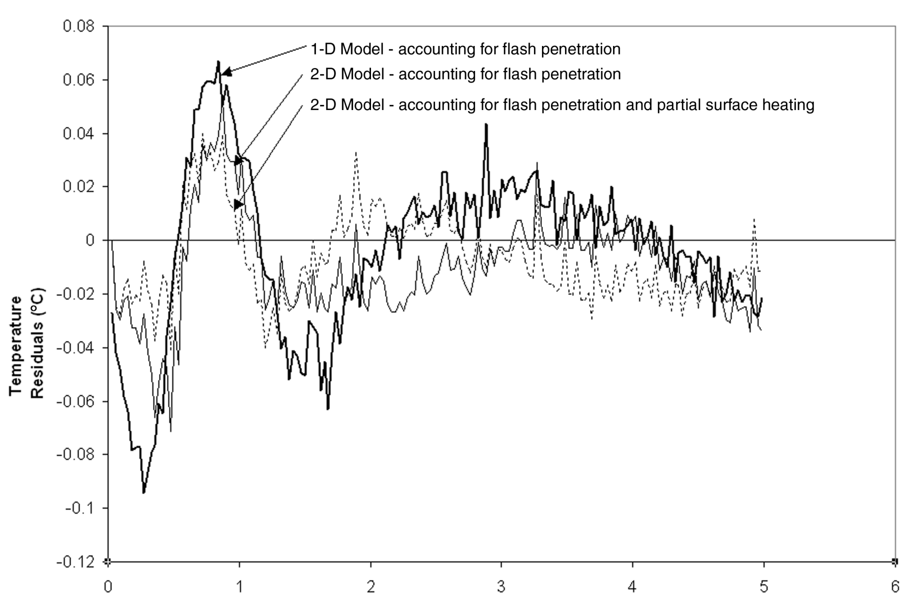

Fig. 4 A comparison of models that account for penetration of the laser flash. Note the significantly smaller temperature scale in comparison to Fig. 3.

Table 2 An application of the *F* test in the three comparisons for which it applies

Comparison	ΔR	$\Delta R/s^2$	F statistic	Additional parameter valid
Models a and c	1.010	843	3.95	yes
Models b and d	0.6297	1272	3.95	yes
Models d and e	0.0184	14.24	3.95	yes

readings could be a slight amount of laser leakage around the edges of the sample in the holder at the instant of the flash.

The "*F*" statistic is a useful and objective means of determining whether the improvement gained in the more complex models with additional parameters is the result of more adequate modeling or simply the result of additional degrees of freedom in the models. In accordance with [12], the *F* test is employed by comparing the sum of squares of the residuals between two models, subtracting one from the other, and designating this difference as ΔR. This term is then divided by the mean sum of squares for the higher-order model s. If this quotient is greater than the *F* statistic for the number of degrees of freedom in the experiment, then the additional parameter in the higher-order model is considered statistically significant to the degree of confidence chosen in selecting the *F* statistic. The most common confidence level used in these types of analyses is the 95% confidence region. Therefore, this level was used in applying the *F* test to the results of the analysis in this research.

The points of applicability of the *F* statistic in this particular analysis are between models a and c, models b and d, and models d and e. This is because, in each of these cases, one additional parameter is being added to the lower-order model, giving it a higher complexity of order one. Correspondingly, the *F* statistic cannot be applied between models a and b or between models c and d because both of these comparisons involve the same number of parameters. Additionally, the *F* statistic cannot be applied between models c and e because an increase in complexity of two orders exists between these models. For the number of parameters and the number of measurements in the experiments analyzed as part of this research, the *F* statistic has a value of 3.95 in each case. Table 2 shows the application of the *F* statistic in the three model comparisons for which the *F* test applies. In each case, the improvement afforded by the additional parameter in the higher-order model proved to be greater than that required for demonstrating statistical significance. Moreover, differences in estimated values of the parameter of interest, thermal diffusivity, of 16.4, 15.0, and 4.0%, respectively, were observed in the three comparisons. Clearly the comparison between models d and e was not as significant as the other two comparisons. However, the addition of the parameter that accounted for the nonuniformity of surface heating was shown to be statistically significant and a valid addition to the model.

Conclusions

Five different mathematical models were tested on laboratory data from a flash diffusivity experiment involving a porous, carbon, solid material. Each successive model produced improved results over its less-sophisticated predecessor. The *F* test was used in providing statistical validation for the improved performance observed in the higher-order models. Models accounting for two-dimensional conduction exhibited superior performance over their one-dimensional counterparts. Models that accounted for penetration of the laser flash into the porous material beyond the surface provided drastically improved conformance to the experimental data when compared with the models that accounted for surface heating only. This was true for both one-dimensional and two-dimensional models.

The model that gave the best overall performance accounted not only for two-dimensional conduction effects and penetration of the laser flash, but also accounted for nonuniform laser heating of the sample surface. In addition to being statistically validated by the *F* test, this model generated results that were 20% different from the one-dimensional model that assumed uniform surface heating.

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